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The Journal *of the* Society of Dyers and Colourists

Volume 75 Number 2

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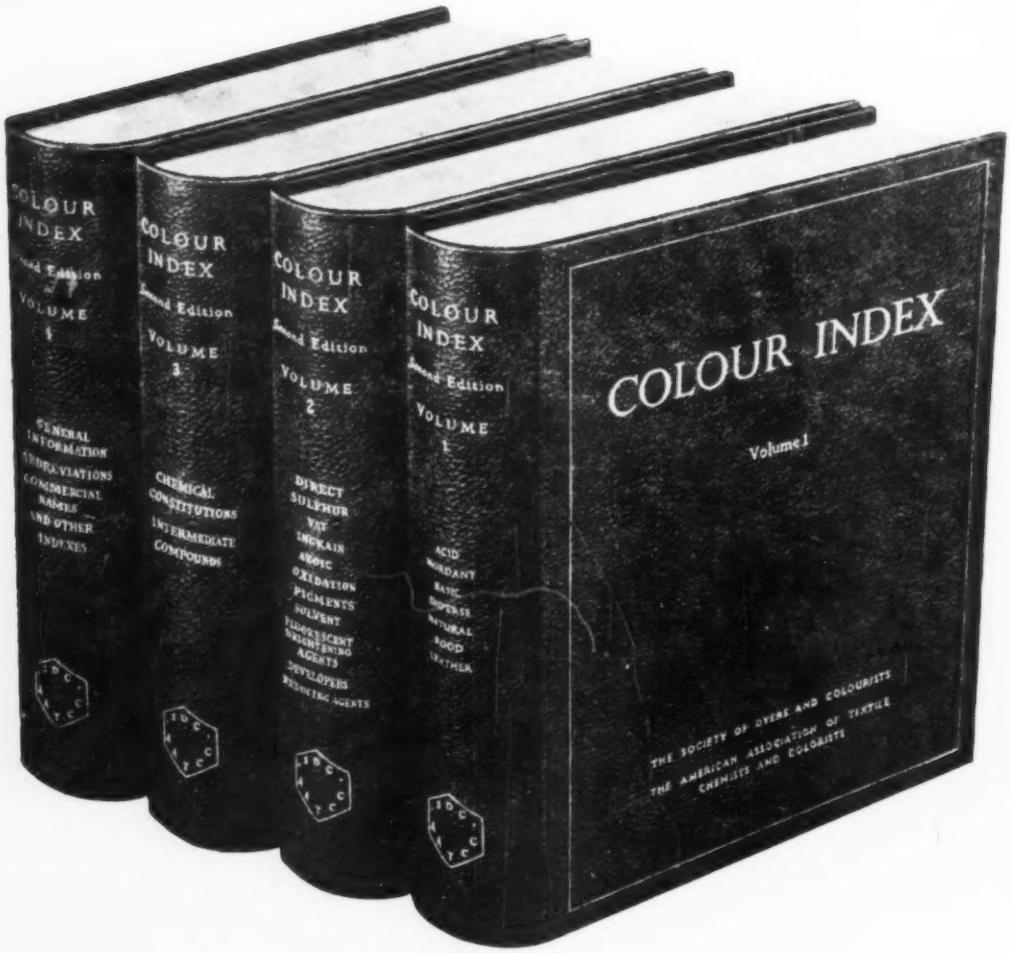

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at **7.30 pm**

by **I. S. MOLL Esq B.Sc**

Title of the lecture

Aspects of Pigment Dispersion in relation to Usage

Members of other societies and the public will be welcome at the lecture

An appeal is made for full support of this important occasion by Members and their friends. All interested are invited to attend

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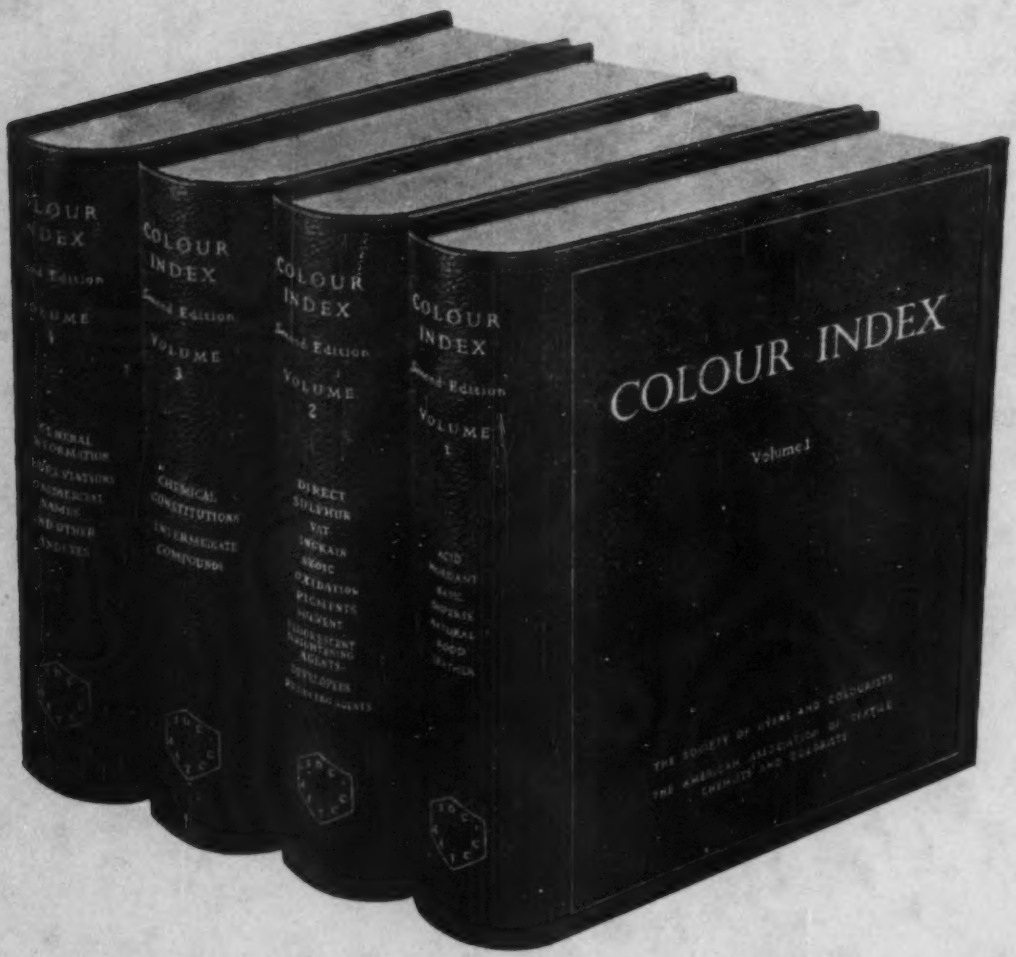

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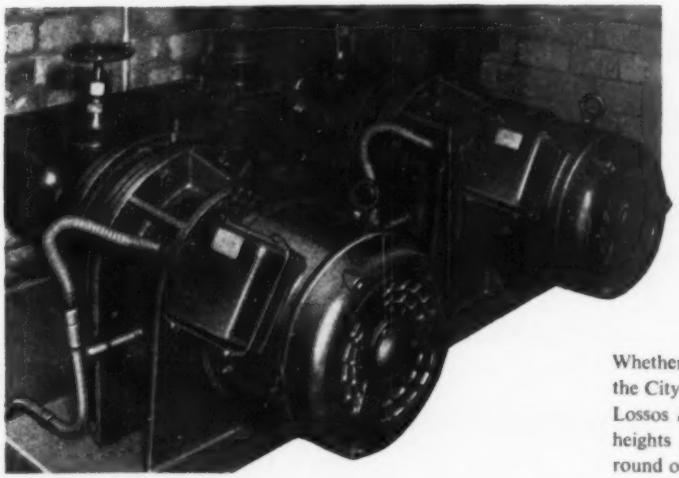

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc, should consult pages 1–10 of the January 1959 and pages 509–516 of the July 1958 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

The Life and Times of Peter Griess	*W. H. Cliffe*
The Mechanism of the Dyeing of Polyester Fibres with Disperse Dyes	*O. Glenz*
Acrylonitrile in the Textile Industry	*J. H. MacGregor*
The Mechanisms of Diazotisation	*J. H. Ridd*

COMMUNICATIONS

Flash Photolysis of Vat Dyes	*N. K. Bridge and W. M. Maclean*
The Supercontraction of Wool Fibres by Aromatic Compounds	*W. G. Crewther*

EXPLANATORY PAPER ON MODERN THEORY

Surface Activity, Solution, and Adsorption	*C. H. Giles*

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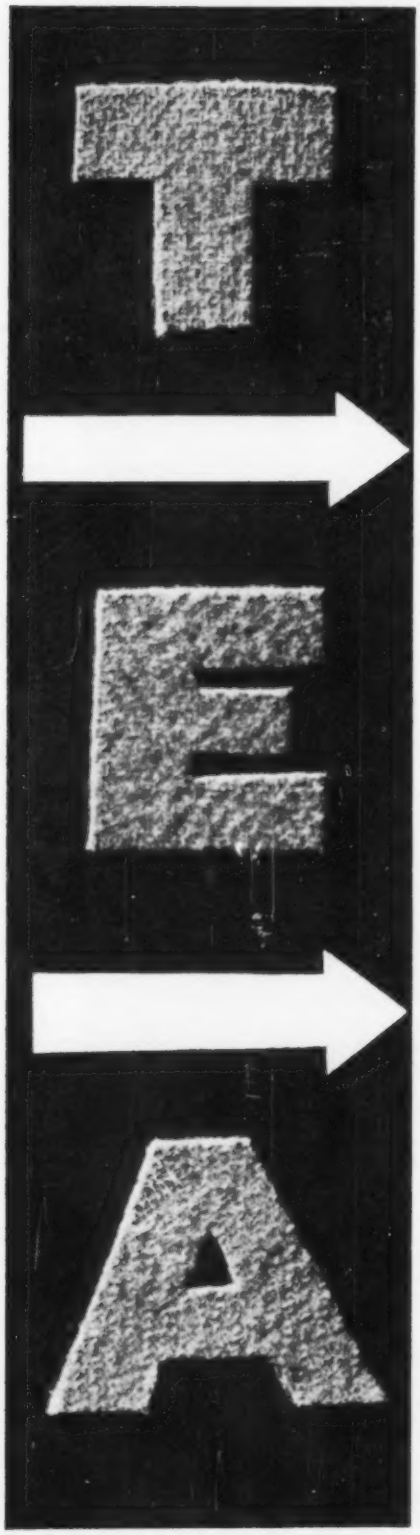

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FORTHCOMING MEETINGS OF THE SOCIETY

Wednesday, 25th February 1959

BRADFORD JUNIOR BRANCH. *Vat Dyeing of Cotton Piece Goods by Pre-pigmentation Routes.* M. R. Fox, Esq., F.T.I., F.S.D.C. and J. F. Mawson, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Institute of Technology, **Bradford.** 7.15 p.m.

Thursday, 26th February 1959

WEST RIDING SECTION. *Fast Shades on Wool-Cellulose Unions.* D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Victoria Hotel, Bridge Street, **Bradford.** 7.30 p.m.

Friday, 6th March 1959

LONDON SECTION. *The Use of Antistatic Agents in Textile Processing.* A. E. Henshall, Esq., B.Sc. (Imperial Chemical Industries Ltd.). The Royal Society, Burlington House, **London W.1.** 6 p.m.

MIDLANDS SECTION. Section Annual Dinner. George Hotel, **Nottingham.**

Tuesday, 10th March 1959

LEEDS JUNIOR BRANCH. *New Methods for the Assessment of Dyeing Properties and Usability of Dyes.* G. H. Lister, Esq., B.Sc., Ph.D. Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, **Leeds.** 3.15 p.m.

NORTHERN IRELAND SECTION. Annual General Meeting. Grosvenor Rooms, **Belfast.** 7 p.m. followed at 8 p.m. by lecture on *Stenter Drying* T. A. Uthwatt, Esq., B.A., A.M.I.E.E., and J. S. Woollatt, Esq., B.Sc. (Mather & Platt Ltd.).

SCOTTISH SECTION. Annual General Meeting. 7 p.m. Followed by *Modern Cloth Drying Principles and Machines.* K. S. Laurie, Esq., A.M.I.Mech.E., A.M.I.E.E. (John Dalglish & Sons Ltd.). St. Enoch Hotel, **Glasgow.** 7·15 p.m.

Wednesday, 11th March 1959

SCOTTISH JUNIOR BRANCH. Annual General Meeting 7 p.m. *Dyeing Unions Containing Acrylic Fibres.* B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. (Ciba Clayton Ltd.). Technical College, **Paisley.** 7.30 p.m.

Thursday, 12th March 1959

BRADFORD JUNIOR BRANCH. *Sizing Materials— Their Chemistry and Application.* J. H. MacGregor, Esq., Ph.D., F.R.I.C., F.C.S. (Courtaulds Ltd.). Institute of Technology, **Bradford.** 7.15 p.m.

MIDLANDS SECTION. *Dyeing and the Future.* J. Boulton, Esq., M.Sc.Tech., F.R.I.C., F.T.I., F.S.D.C., President of the Society. (Joint meeting with the Coventry Textile Society.) Courtauld's Acetate and Synthetic Fibres Laboratory, Lockhurst Lane, **Coventry.** 7 p.m.

SCOTTISH SECTION. *The Reactivity of Keratin.* Professor J. B. Speakman, D.Sc., F.R.I.C., F.T.I., F.S.D.C. Scottish Woollen Technical College, **Galashiels.** 7.30 p.m.

WEST RIDING SECTION. *The Sorption of Moisture by Fibre-forming Polymers.* L. Valentine, Esq., B.Sc., Ph.D. (Tootal Broadhurst Lee Co. Ltd.). Victoria Hotel, Bridge Street, **Bradford.** 7.30 p.m.

Saturday, 14th March 1959

BRADFORD JUNIOR BRANCH. Annual General Meeting. Institute of Technology, **Bradford.** 10.15 a.m.

Tuesday, 17th March 1959

HUDDERSFIELD SECTION. *Fast Shades on Wool-Cellulose Unions. Recent Advances in this Field.* D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Silvios Cafe, **Huddersfield.** 7.30 p.m.

Wednesday, 18th March 1959

MIDLANDS SECTION. *Photoelectric Colorimeters . . . their uses and limitations in the Dyehouse.* J. V. Alderson, Esq., B.Sc., A.R.C.S. (Joint meeting with the British Association of Chemists.) Midland Hotel, **Derby.** 7 p.m.

Wednesday, 18th March 1959

NORTHERN IRELAND SECTION. Annual Dinner. Grosvenor Rooms, **Belfast.** 7 p.m. for 7.30 p.m.

Thursday, 19th March 1959

MANCHESTER JUNIOR BRANCH. Films— *Silk. Principles of Chromatography. How the World Looks to a Colour Defective.* Room J/B 16 — Chemical Engineering Building (Jackson Street), College of Science and Technology, **Manchester.** 4.30 p.m.

Friday, 20th March 1959

MANCHESTER SECTION *Symposium on Reactive Dyes.* College of Science and Technology, **Manchester.**

Afternoon Session, Lecture Theatre (Room F.1). 4 p.m. to 5 p.m. *The Reactions of Procion Dyes with Cellulose.* A. S. Fern, Esq., B.Sc., D.I.C., F.S.D.C. (I.C.I. Ltd., Dyestuffs Division, Manchester). 5.15 p.m. to 6.30 p.m. Interval for Tea.

Evening Session, Great Hall (Room C.1). 6.30 p.m. to 7.30 p.m. *Some Relations between the Chemical Constitution of Cibacron Dyes and their Dyeing Characteristics.* Dr. J. Wegmann (Ciba Limited, Basle). 7.30 p.m. to 8.30 p.m. *The Remazol Linkage.* Dr. E. Bohnert (Farbwerke Hoechst). 8.30 p.m. to 9 p.m. Discussion.

Thursday, 2nd April 1959

MIDLANDS SECTION. *Colour Physics and Match Prediction.* (Lecturer to be announced later.) (Joint meeting with the Textile Institute.) Carpet Trades Ltd. Canteen, **Kidderminster.** 7 p.m.

Friday, 3rd April 1959

LONDON SECTION. *Recent Developments in Dyeing.* T. Vickerstaff, Esq., M.Sc., Ph.D., F.S.D.C. (Imperial Chemical Industries Ltd.). George Hotel, **Luton.** 7 p.m.

Thursday, 9th April 1959

WEST RIDING SECTION. Annual General Meeting. Colour Film on *How the World Looks to a Colour-defective.* Victoria Hotel, Bridge Street, **Bradford. 1**

Friday, 10th April 1959

The Society's Annual General Meeting and Dinner, Grand Hotel, **Leicester.**

Wednesday, 15th April 1959

MIDLANDS SECTION. Annual General Meeting of the Section followed by a showing of New Scientific Films. King's Head Hotel, **Loughborough.** 7 p.m.

Thursday, 16th April 1959

MANCHESTER JUNIOR BRANCH. Three short papers by members of the section. Room J/B 16 — Chemical Engineering Building (Jackson Street), College of Science and Technology, **Manchester.** 4.30 p.m.

Friday, 17th April 1959

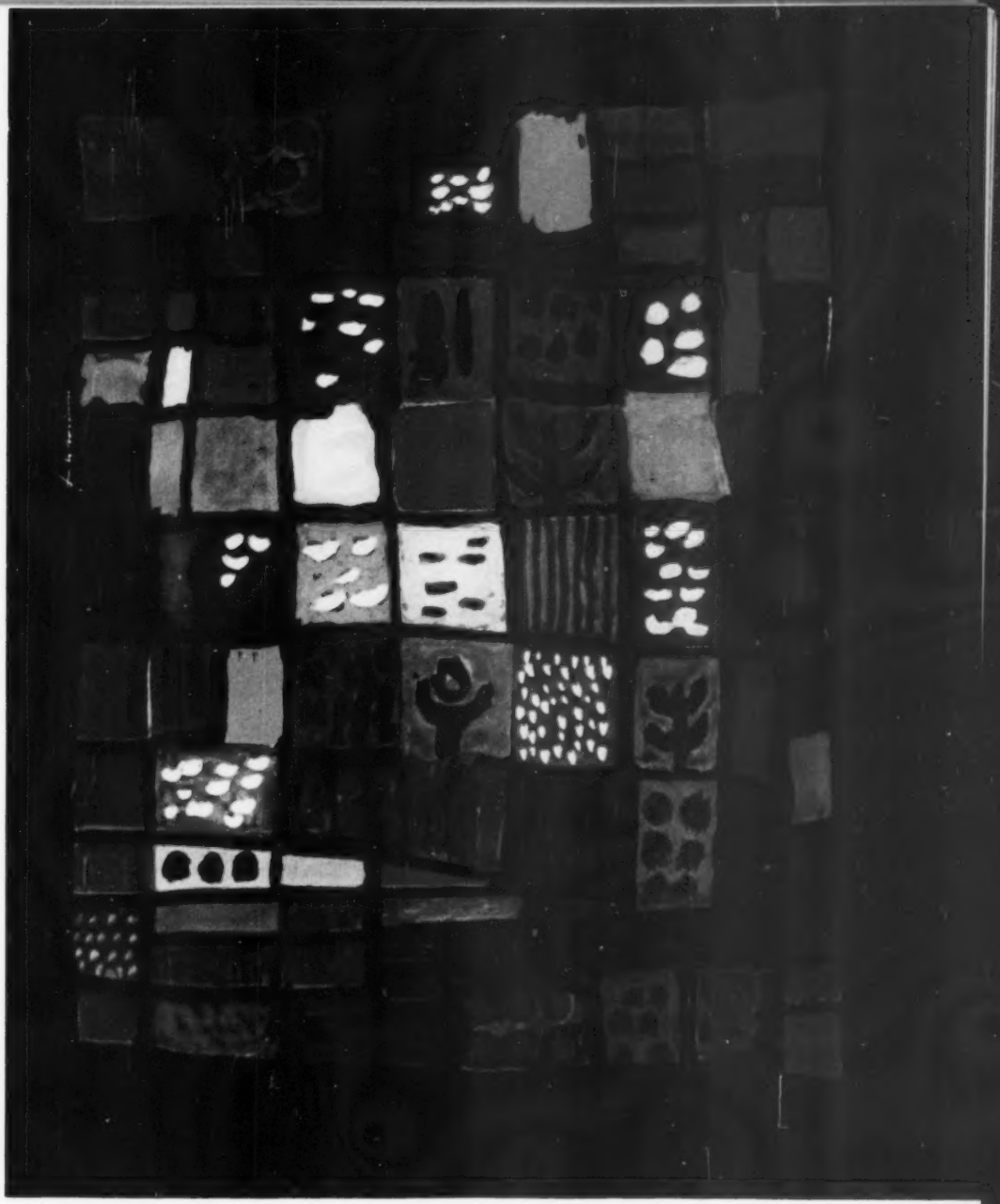
MANCHESTER SECTION. Annual General Meeting. *Stenter Drying.* A. T. Uthwatt, Esq. (Mather & Platt Ltd., Research Dept.). The Textile Institute, **Manchester.** 7 p.m.

Tuesday, 21st April 1959

HUDDERSFIELD SECTION. Annual General Meeting. Followed by a Colour Film on *How the World Looks to a Colour-defective.* Cmdr. Dean Farnworth (United States Naval Research). Silvios Cafe, **Huddersfield.** 7.30 p.m.

Friday, 24th April 1959

LONDON SECTION. Annual General Meeting. Commander Dean Farnworth (United States Navy) will show and discuss his film *How the World Looks to a Colour-defective.* Waldorf Hotel, **London W.C.2.** 6 p.m.



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THE JOURNAL

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FEBRUARY 1959

Proceedings of the Society

THE THIRTY-GEORGE DOUGLAS LECTURE

Textile Finishing
Problems of Today and Tomorrow

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THE JOURNAL

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Volume 75 Number 2 FEBRUARY 1959 *Issued Monthly*

Proceedings of the Society

THE FIFTH GEORGE DOUGLAS LECTURE

Textile Finishing
Problems of Today and Tomorrow

WALTER GUTMANN

Meeting held at the Midland Hotel, Bradford, on 5th November 1958, Mr. John Boulton (President of the Society) in the chair

On the basis of the four fundamental requirements of textiles — sales appeal, wear resistance, ease of maintenance, and body protection — a survey is given of the resulting problems for the textile finisher. The contributions of chemistry and application development as well as recently of physics, mathematics, and electronics to modern finishing techniques are demonstrated by several examples with textiles of natural and synthetic fibres. The results obtained and possible achievements of future research work are considered. The present outstanding task is seen to lie in gaining precise and complete knowledge of the interrelationship between garment and human body to provide maximum comfort in clothing for any type of use by adjusting the fabrics to the necessities of body comfort.

INTRODUCTION

When, at the beginning of this year, I received the invitation to deliver the Fifth George Douglas Lecture on the subject of *Textile Finishing* I felt not only greatly honoured but also conscious of the considerable responsibility which this task entailed and which was now imposed upon me. A close study of the relevant literature made me realise more and more how very closely Mr. George White[1] had expressed my own feelings when, in his introductory remarks to the Fourth George Douglas Lecture on *Dyeing*, he spoke of his great reservations and his doubts whether he would be able to present a paper worthy of the memory of George Douglas, bearing in mind the vast number of publications already existing on this subject.

The results which have been achieved in dye chemistry are quite evident to us all and can be perceived by our unaided eyes. No other sense is able to provide us with so clear an analysis as the eye achieves in respect of colour. Nearly a hundred years of dye chemistry had to elapse before physicists were able to find a scientific method of measuring objectively the subjective impression of the human eye, namely by reflection photometry. Nowadays, we know that there exists not only a nerve track from the eye to the visual centre, but also a second track which leads to the hypophysis cerebri, where colours induce in us active or passive behaviour; that is to say, they not only appeal to our sense of beauty but also influence our attitude to life. Finally, I may point out that it was dye chemistry which, as a result of its research, has provided the basis for the giant structure of modern organic chemistry.

Considering the proud achievements of the dye chemist, the textile finisher faces a difficult task in gaining his share of credit for his efforts and successes. I should like to say — if I may repeat that exceedingly well chosen metaphor of Mr. White's — that I am not at this moment in the position of a man who, after climbing the top of a hill, is able to describe the scenery surrounding the peak. That mountain I am talking of is still to be conquered. I consider it my task to inform you of the altitude we have reached and of the possible paths towards the summit.

OBJECTS OF TEXTILE FINISHING

The finish of textiles is a matter of vital interest these days. I need only remind you of the labels with which modern textiles are provided, testifying, for instance, to water-repellency, mothproofing, shrink-resistance, ease of laundering and drying, or permanent pleating. Textiles labelled in this way may today become best-sellers. But publicity for publicity's sake does not in the long run give us what we want. Short-lived sales gimmicks succeed only in dissipating our efforts. Of course, finishers must keep abreast of the latest developments in fashion and technique, for after all we want to sell

our goods. Our task, however, is bigger than this: we must get away from mere advertising by actually educating ourselves and our customers, and we must provide our goods with additional qualities which grant the wearer lasting pleasure and comfort, in other words with qualities that enrich his life.

Our language, as so often, contains an indication of our ambition. The English word "finish" is a short and precise one. Hearing this word, we think of cloth receiving the last touch, so that it has a pleasant handle and drape, an appealing sheen, etc., in the same way as the word "finish" is used for painting or polishing. The German word for finish is *ausrüsten*. This may mean very little to the non-professional, as the word does not say anything about the work it describes. It means "to provide necessities", and its English equivalent would be "to equip", say a ship or an army, that is to say to give them what they need, in order to withstand the demands that they may encounter on the way. It is our problem now, by applying our techniques, to provide the two effects indicated by the two words, namely the finish and the equipment. To do this we must incorporate four basic qualities into our textiles—

(1) HIGH SALES APPEAL based on attractive handle, draping, appearance, and suppleness.

(2) HIGH WEAR RESISTANCE as the result of—

- (*a*) Adequate tensile strength, and abrasion and weather resistance
- (*b*) Dimensional stability, crease resistance, and freedom from pilling
- (*c*) Special effects, such as water repellency, antistatic behaviour, reduced flammability, mothproofing, odour repellency, etc.

(3) EASE OF MAINTENANCE, because of reduced tendency to soiling and effortless cleaning.

(4) OUTSTANDING BODY PROTECTION as a result of proper heat insulation, moisture absorption and transmission, and adequate air, gas, and light permeability.

I have deliberately listed this detailed catalogue of requirements at the beginning of my paper. We know that at the present stage of development some of these problems have been mastered, and others can be overcome only by means of a compromise, but in the case of many we are still groping towards a solution. In this paper, I should like to take you through this variety of problems. We shall be able to follow only the main lines and to dwell upon a few major problems. In doing so, I must thank the many inventors, research workers, and authors who have provided such signposts as there are. I hope that you will understand why I do not mention their names except in the printed references and why I refrain from calling many valuable products by trade names and use their chemical descriptions only: it will save time and will also be clearer.

FINISHING TECHNIQUE TODAY

Finishing has originated from an empirical art, many centuries old. We have to admire the astonishing dexterity and the intuitive skill of our ancestors, who were forced to obtain the finish of their fabrics with simple machinery and a very restricted range of chemical auxiliaries. How close to our aim were they? Considering only five of the above-mentioned requirements— handle, crease resistance, heat insulation, moisture absorption, washability— we know that a wool fabric meets the first four requirements, a cotton fabric only the last two. Thus, the textile finisher was left with the inherent properties of his fibres.

Three events provided the impetus for the development of the original craftsmanship into the present-day techniques—

(1) The introduction of chemistry into the field of textile finishing, which has enabled us to provide textiles with properties not intrinsic to the fibre.

(2) The large-scale use of regenerated cellulose fibres, which compelled us to develop processes now known as *resin finishing* and which gave rise to the modern application techniques in textile finishing.

(3) The large-scale use of synthetic fibres, which, besides chemistry, brought into play physics, mathematics, electronics, and engineering, and which, incidentally, is placing the finisher in the position of arbiter or, as I should prefer to say, mediator in the conflict between natural and synthetic fibres.

CHEMISTRY IN TEXTILE FINISHING

The chemistry of textile finishing, too, had its origin in dyeing problems. That is the common factor between the dyer and the finisher. To give an idea of the tremendous contributions of synthetic chemistry, let me cite three striking examples.

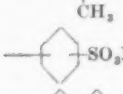
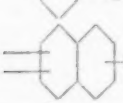
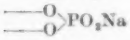
The first products were surface-active agents, such as wetting agents, dispersing agents, emulsifying agents, levelling agents, and, of course, detergents. In the period between the First and Second World Wars basic knowledge concerning polar structure, mode of action, and balance between lipophilic groups and hydrophilic groups was unravelled, knowledge which has resulted in our present broad spectrum of surface-active materials (Table I)[1a]. Developments in this field are largely complete, and any possible further improvements can be safely pursued at leisure. The main emphasis, therefore, centres on supporting the chemically produced surface forces by physical means, i.e. by construction of efficient machines.

It is remarkable that the surface-active products which we have developed for the textile industry were, after the Second World War, used on a much larger scale in other industries, first of all in the U.S.A., and now practically everywhere else. They are used, for instance, in the detergents industry in powder or liquid form and recently even in the form of soap bars; in cosmetics they find application as detergents and emulsifiers; in the petroleum industry they are used as demulsifiers and additives, and to improve yield; in the building industry they serve as additives to

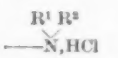
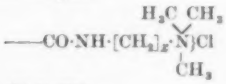
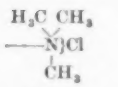
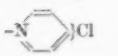
TABLE I

Surface-active Agents

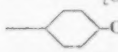
ANION-ACTIVE

——COONa	Soap
——CO·N(CH$_3$)·CH$_2$·COONa	Acylsarcosinate
——CO·[NH·CH(R)·CO]$_x$·NH·CH(R)·COONa	Acyl peptide
——O·SO$_3$Na	Primary alkyl sulphate (sulphated fatty alcohol)
——O·[C$_2$H$_4$·O]$_x$·SO$_3$Na	Alkyl polyether sulphate
——CH(O·SO$_3$Na)——	Secondary alkyl sulphate (sulphated olefin)
——CH(SO$_3$Na)——	Secondary alkyl sulphonate
——COO·C$_2$H$_4$·SO$_3$Na	Acylisethionate
——OOC·CH$_2$	Dialkyl sulphosuccinate
——OOC·CH(SO$_3$Na)·CH$_2$·COO——	
——CO·N(CH$_3$)·C$_2$H$_4$·SO$_3$Na	*N*-Acyl-*N*-methyltaurate
	Alkylbenzenesulphonate
	Dialkylnaphthalene-sulphonate
	Dialkyl phosphate

CATION-ACTIVE

	Alkylamine hydrochloride
	Alkylaminoamide hydrochloride
	*N*-Alkyltrimethyl-ammonium chloride
	Alkylpyridinium chloride

NON-IONIC

——COO·[C$_2$H$_4$·O]$_x$·H	Polyoxyethylene fatty-acid ester
——CO·N<[C$_2$H$_4$·O]$_x$·H, [C$_2$H$_4$·O]$_y$·H	Polyoxyethylene fatty amide
	Alkylphenol polyoxyethylene ether
——O·[C$_2$H$_4$·O]$_x$·H	Alkyl polyoxyethylene ether
——N<[C$_2$H$_4$·O]$_x$·H, [C$_2$H$_4$·O]$_y$·H	Polyoxyethylene fatty amine

concrete; in the metal industry they find application in cleaning and flotation; in agriculture they play their part as emulsifiers for insecticides; and, finally, in modern dry cleaning they appear as intensifiers to remove water-soluble soil. Not including products for special purposes, the alkylbenzenesulphonates, as the least expensive products, have the largest turnover. Sulphated and sulphonated aliphatic compounds such as fatty alcohol sulphates or fatty acid condensation products occupy an intermediate position, while the consumption of the most efficient surface-active compounds, the non-ionics, shows everywhere a strong upward trend— a picture which reflects to a large extent their position in the textile industry [2]. Such an abundance of products as is provided by chemistry in this field has inevitably led to the necessity for finding a proper balance between price and efficiency, a situation which might encourage the imagination of the consumer but baulks that of the man in the laboratory.

TABLE II

Durable Stiffening Agents

–[CH$_2$–CY(R)–CH$_2$–CY(R)]$_n$–

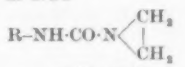
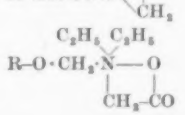
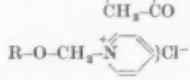
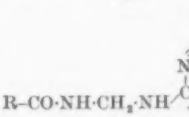
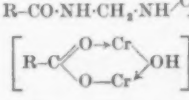
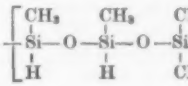
Y	R	
H	Cl	Polyvinyl chloride
H	O·CO·CH$_3$	Polyvinyl acetate
H		Polystyrene
H	O·C$_n$H$_{2n-1}$	Polyvinyl ethers
H	COOR	Esters of polyacrylic acid
H	O·R^2·O (=2R)	Polyvinyl acetals
CH$_3$	COOR′	Esters of polymethacrylic acid

–[O·CO·NH·R·NH·CO·O·R′]$_n$– Polyurethanes

The problem of a permanent increase in stiffness and body of textiles has likewise been solved [3]. The potentialities range from the use of sulphuric acid, cuprammonium solution, and ethylene oxide, from the fixation of water-soluble finishes, e.g. starch, gelatin, or polyvinyl alcohol, with formaldehyde resins or ethyleneimine compounds, to the use of insoluble thermoplastic or thermosetting resins (Table II) or rubber. Even wool textiles, which appear to object, when treated with a filling finish, welcome plastic dispersions such as polyvinyl acetate as long as the latter are highly disperse. The discovery of these numerous types of plastics applicable to textiles has created new techniques also for this sector, such as lamination or coating of textiles with plastic emulsions or films, and the bonding of loose fibres to form non-woven fabrics [4, 5]. These processes overlap the boundary

TABLE III

Waterproofing Agents

R–NCO	Stearyl *iso*cyanate
	Alkylethyleneimineurea
	*NN*-Diethylbetaine ether
	Pyridinium chlorides
	Water-repellent melamine resins
	Chromium–stearic acid complex
	Silicone resins

between textile and plastics technology and represent a matter of vital industrial importance.

Finally, the old and yet always topical problem of water-repellent finishing has been solved satisfactorily. The products applied mostly are paraffin-wax emulsions containing aluminium or zirconium salts. By using stearic acid derivatives [5] (Table III) or silicones [6, 7] we have succeeded in achieving water-repellent finishes with a soft, smooth handle, in waterproofing synthetic fibres, and in combining water repellency with crease resistance [8]. Although the old problem of providing a really durable water repellency has remained unsolved, the excellent waterproofing agents that are now available to the dry cleaner have robbed the problem of most of its urgency.

I hope that these three examples, chosen at random from among the laurels earned by chemical research, have sufficiently indicated the outstanding contribution that chemistry has made to the problem of textile finishing. Let me now deal with three problems which are yet to be solved.

The first and long-standing problem is the softening of fabrics of natural or synthetic fibres [2, 9, 10]. None of the many softeners successfully used on regenerated cellulose fibres (Table IV) show the desired result on wool or synthetic fibres, and on cotton only if suitable calendering treatment is applied. These auxiliaries, therefore, do not act as plasticisers, but rather as lubricants. In this capacity they eliminate the stiffness of dried rayon and the surface roughness of cotton. Thus we are left with the use of fine fibres and suitable construction of our yarns and fabrics.

TABLE IV

Softening Agents

ANION-ACTIVE	
$\text{——COO–CH}_2\text{——COO–CH——COO–CH}_2\text{——}$ $\text{O·SO}_2\text{Na}$	Sulphated oil, sulphated fat
$\text{——CO·N·CH}_2\text{·COONa}$ CH_3	Acylsarcosinate
SO_2Na	Sulphonated paraffin
CATION-ACTIVE	
$\text{——N}\langle^{\text{R}}_{\text{R'}}\text{,HX}$	Fatty amine salt
$\text{——N}^{+}\langle^{\text{R}}_{\text{R'}}\rangle\text{X}^{-}$ R''	Quaternary ammonium salt
$\text{——CO–N–C}_2\text{H}_4\text{–N–C}_2\text{H}_4\text{–N}^{+}(\text{CH}_3)_3\text{X}^{-}$ $\text{CH}_3 \quad \text{CH}_3$	Quaternary ammonium salt of aminoamide
$\text{——COO·C}_2\text{H}_4$ $\text{X·O·C}_2\text{H}_4\text{–N,HCl}$ $\text{X·O·C}_2\text{H}_4$ $\text{X = H or [C}_2\text{H}_4\text{·O]}_x\text{·H}$	Salt of aminoester
$\text{——C}\langle^{\text{N–CH}_2}_{\text{N–CH}_2\text{,HX}}$ H	Salt of iminazoline
$\text{——CO·NH·C·NH·C·NH}_2\text{,2HX}$ $\text{NH} \quad \text{NH}$	Salt of diguanide
NON-IONIC	
$\text{——O·[CH}_2\text{·CH}_2\text{·O]}_x\text{·H}$	Alkyl polyoxyethylene ether
$\text{——N}\langle^{\text{[CH}_2\text{·CH}_2\text{·O]}_x\text{·H}}_{\text{[CH}_2\text{·CH}_2\text{·O]}_y\text{·H}}$	Polyoxyethylene fatty amine
$\text{——COO·[CH}_2\text{·CH}_2\text{·O]}_x\text{·H}$	Polyoxyethylene fatty-acid ester
$\text{——CO–N}\langle^{\text{[CH}_2\text{·CH}_2\text{·O]}_x\text{·H}}_{\text{[CH}_2\text{·CH}_2\text{·O]}_y\text{·H}}$	Polyoxyethylene fatty amide

The two other problems, namely antistatic and dirt-repellent finishes, were presented to chemistry as a result of the large-scale use of synthetic fibres. In the competition between cotton and synthetic fibres, these two problems are of decisive importance.

Electrostatic charging is caused by separation and friction phenomena on all objects, and therefore also on textiles. The magnitude and the sign of the charge are dependent on accidental circumstances and are rather capricious. In the case of electric conductors, the charge, of course, is weak as a result of charge neutralisation, but it is nevertheless present. One single electron on a surface of 10^{-12} sq.cm. is sufficient to produce the highest amount of static charge experienced on textiles [11].

TABLE V

Electrostatic Series at 15% R.H. [12]

Wool	+ *positive*
Nylon	
Silk	
Viscose rayon	
High-tenacity viscose rayon	
Cotton	
Fiberglas	
Spun ramie	
Cellulose acetate	
Polyester fibre	
Acrylic fibre	
Polyethylene	
Saran	— *negative*

There are two ways of reducing static charging in textiles—

(1) By placing filaments of different charge (Table V), e.g. polyamide and polyester fibres or filaments, side by side [12], a method which has in fact been applied. However, it will be successful only if the components show absolutely constant behaviour under all conditions [13, 14].

(2) By making insulating filaments conducting either through the admixture of conducting filaments or by applying an antistatic finish [15]. The first method, the mixing of fibres, has been successfully developed. The chemical treatment, i.e. antistatic finishing, is satisfactory only with compounds that are not resistant to washing. Of the so called permanent finishes known today, only a few are really resistant to all types of detergents, fatty alcohol sulphates included. All these products provide the fibres with a coating and, therefore, with an undesirable handle, and none of them are effective under Arctic conditions.

Much has been written about the soiling of natural and synthetic fibres. Soil usually consists of dust particles and fatty materials. Even in the

case of synthetic fibres, which are normally considered to be smooth, irregularities may be observed under the microscope at a magnification of 10,000–20,000, particularly after long wear. These irregularities are able to harbour even the smallest dust particles [16]. This is the reason why a textile is subject to fairly rapid, stubbornly retained soiling. This tendency may be counteracted by filling the uneven parts of the fibre surface with very fine silica or alumina gel or with suitable polymers, e.g. stearylethyleneimineurea [17]. Recently an oil-repellent finish has come on to the market which is based on perfluorinated compounds [18]. No finish is as yet available to repel both oil and pigment particles simultaneously. This is a gap in finishing which is still delaying the large-scale use of synthetic fibres in "minimum-care" articles, e.g. men's shirts.

Recently published results on the graft-polymerisation of fibres in fabrics under the influence of ionising radiation seem to be promising for the solution of both problems [19]. At first glance, such a method appears difficult to achieve in large-scale production. However, it may well be possible by this method to graft polymers of any type upon the surfaces of fibres and thereby alter at will surface properties of fabrics such as roughness or conductivity.

We can always rely on organic synthetic chemistry to supply us with the key tools for our work. This is again illustrated by one of the latest achievements, the deodorising finish, which may become a welcome improvement, e.g. for stockings and perhaps underwear. Organic tin compounds are particularly suitable for this purpose on account of their bactericidal and fungicidal properties [20, 21].

APPLICATION TECHNIQUES IN TEXTILE FINISHING

The large-scale use of regenerated cellulose fibres has led to finishing processes in which the technique of application is at least as important as the chemical aspect. These are the swelling- and crease-resistant finishes [3, 10]. As these processes today, some thirty years after their introduction, have again become subjects of vital technical and economic interest, I should like to dwell upon them for a few moments. The three basic patents [22, 23], two of them British, one German, still describe correctly the chemical fundamentals, even nowadays, when the range of suitable chemical compounds has greatly widened. Moreover, our knowledge of what happens in a fabric is more complete and precise. We know now that we do not in fact make the fibres crease-resistant. A crease-resistant fibre would be a completely elastic fibre, such as a rubber fibre, for example. Fabrics made from such fibres would indeed not have the character of a textile: they would neither drape well nor would they retain their shape or creases. We know that we increase the form stability of the fibres, and consequently all crease-resistant finishes may, with slight modifications, also be used for embossing and for gloss effects.

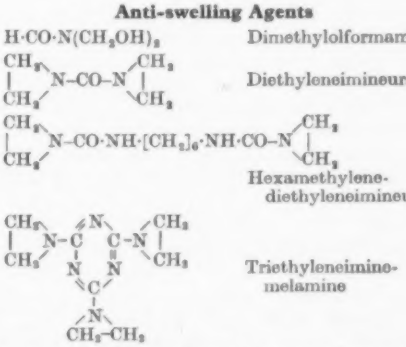
This is also the reason why among the synthetic fibres it is not the highly elastic ones but the form-stable or shape-retaining fibres that give the best crease-resistant fabrics.

On account of this fact, we must arrange the fibres in the yarn and the yarn in the fabric in such a way that creasing causes little fibre deformation and allows the fibres to return to their original position in the yarn and the fabric respectively. This means that fibres should be distributed at random three-dimensionally and not held too tightly in their position. In this respect we can learn much from the manufacturer of woollen fabrics, who obtains the required arrangement of the fibres by traditional methods. This is favoured by the inherent properties of the wool, such as high and water-resistant crimp and its capacity to be felted and steam-set.

With spun rayon staple fabrics a similar success has been achieved by preswelling the fabric followed by controlled shrinkage, in other words by complete relaxation. Nowadays, when we know the sensitivity of the process of incorporating resins, the significance of pH control, i.e. the function of the catalyst, the danger of migration at fabric humidities down to 30%, the occurrence of shrinkage below this point, in short how to master that process, we are able to impart good crease-resistant finishes to spun rayon fabrics.

TABLE VI

Anti-swelling Agents

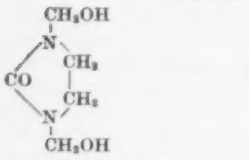


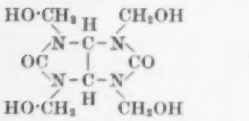
Much more difficult with these fabrics is the production of a durable finish which is fast to repeated laundering. During the Second World War such processes had to be performed on a very large scale on the Continent. Incorporation of resins does not provide adequate wash resistance: the cellulose chains must be chemically cross-linked [24]. For this purpose a large number of compounds have been used (Table VI). Unfortunately, all of them need such an uncommon amount of care in the close observation of processing conditions to enable us to steer a safe course between the Scylla and Charybdis of this finish—fibre embrittlement and inadequate reduction of swelling— that, after the war, when cotton was again available for that type of fabric, these finishes were abandoned at once, almost everywhere.

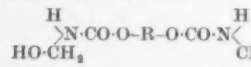
Finishers did not for a very long time dare to apply the crease- and swelling-resistant finishing methods to cotton, a reluctance which can well be appreciated. Regenerated cellulose fibre consists of 50–80% amorphous portions, whereas cotton has a crystallinity of 50–80%. While in the case of regenerated cellulose fibre the amorphous as well as the crystalline constituents are able to take

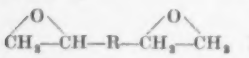
up water-soluble precondensates, the crystalline parts of cotton are inaccessible to water and, therefore, to the finishing agent.

TABLE VII
Reactants


Dimethylolethyleneurea (D.M.U.)


Tetramethylolacetylenediurea


Diurethane

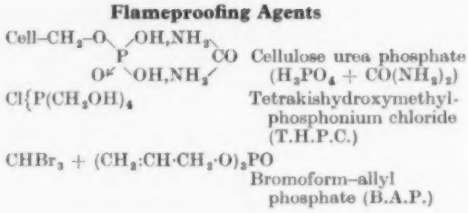

Epoxy resins

The success of the synthetic fibres right from the word "go" in the creation of "wash-and-wear" styles and the enthusiastic reaction on the part of buyers to the possibility of more effortless washing finally forced the adoption of these processes for cotton goods. By mercerisation or, better still, tension-free pretreatment with caustic soda [25] or nitric acid, it became possible to render cotton more amenable to synthetic-resin precondensates. It was recognised not only that yarn and fibre structure are decisive for success but also that not every type of cotton is equally suitable for this treatment. The wash resistance of urea– or melamine–formaldehyde resins is not sufficient for "wash-and-wear" articles. Moreover, their characteristic property of retaining chlorine, because of their nitrogen content, leads to yellowing. For this reason, many new compounds (Table VII) were used which react with the cellulose itself and which do not act primarily as resin-forming substances. Among these, the cyclic urea compounds [26] predominate at present. The epoxy resins, being free from nitrogen, are even less affected by chlorine aftertreatment [27].

In spite of the present tremendous economic success of these processes, we must continue to foster future developments. The price that we pay in terms of reduced strength and flexibility is undesirably high. The roughness of the fabrics occasionally leads to skin irritation. Static charging and tendency to soiling are similar to those experienced with synthetic fabrics. Much, therefore, still needs to be done.

Since the summer of 1958 a method has been introduced in the U.S.A. that provides the desired wet crease-resistance solely through cross-linking, thus avoiding the disadvantages of resin incorporation [28]. I have not yet had any personal experience of this method. It is to be hoped that it will be possible to produce a method applicable under factory conditions for controlling this certainly quite difficult reaction, which may lead to further developments in this field.

TABLE VIII
Flameproofing Agents


Cellulose urea phosphate ($H_3PO_4 + CO(NH_2)_2$)

Tetrakishydroxymethylphosphonium chloride (T.H.P.C.)

Bromoform–allyl phosphate (B.A.P.)

Among related topics there is the durable flame-resistant finish in which phosphonium compounds (Table VIII) are firmly anchored by means of thermosetting resins [29, 30]. The very difficult technique applied in these finishing processes would not have been evolved without the work previously done on crease-resistant finishing.

Finally, I have in mind the chemical finishing of wool. It is significant that the fundamental knowledge provided by British scientists [31, 32] of the structure and behaviour of wool is now finding practical application in the chemical finishing of this complicated natural fibre: apart from the numerous processes to render wool free from felting, methods are now being developed to create durable creases in wool fabrics by fixation with thioglycollic acid [33] or by cross-linking its molecules through the action of alkalis [34, 35].

PROBLEMS IN THE FINISHING OF SYNTHETIC FIBRES

Synthetic fibres, whose properties promise to open up a completely new vista of textile developments, present dyer and finisher with unexpected problems. Today, when we are able to produce from synthetic fibres light-weight materials of great elegance as well as bulky fabrics of extreme softness, is perhaps the right moment to recollect the original disappointment with fabrics made from synthetic yarns, for instance the film-like handle so unlike textiles, the poor draping and resilience, the dreaded pilling effect. By overcoming these disadvantages, fibre producers have earned our gratitude for their contributions to textile technology as a whole, even if made for their own benefit.

We have been able to improve decisively the handle of synthetic textiles by using fibres with the correct denier and by suitable blends of denier and shrinkage, by peeling off the surface layer, as can be done with polyester fibres [36], by the durable embossing of very fine, hardly visible designs, and by complete relaxation of the fabric. A promising contribution to this problem has been made by American scientists, who have reduced the possible errors arising from subjective assessment by the employment of electronic computers. They determined, for instance, the mathematical rank correlations, i.e. the most probable order of quality or, on the basis of the modulus of torsion and bending and relaxation behaviour, the handle likely to be obtained [37, 38]. What has yet to be accomplished is to render the surface of the individual fibres or filaments less smooth, that is to say to make it more similar to that of the natural fibres.

We have moderated the pilling problem by anchoring the high-tenacity fibres so firmly into

the yarn that they do not migrate outwards. For this purpose a whole series of improved machines has been used— high-temperature pin stenters for setting with controlled shrinkage, shock thermo-setting machines for unusually high setting temperatures[39], highly efficient singeing, brushing, and shearing machines, etc. Our desire to produce from synthetic fibres yarns and fabrics of open texture and free from pilling has not yet been satisfied.

Our most interesting task at present is to find out how we can utilise to the full the excellent shape retention of our fibres for the achievement of crease-resistant fabrics. The knowledge gained in the finishing of fabrics from wool and cellulose fibres applies also in this case. But with these swelling-resistant fibres it is far more difficult to achieve irregular distribution in the yarn and complete relaxation of the fabrics. Shrinkage is induced by heat or by mechanical or chemical means. The first method is the one normally used in practice. By being able to shrink synthetic fibres with heat, considerable progress has been achieved with respect to improved handle, bulkiness, and crease-resistance, especially, if fibres of various shrinkage characteristics are blended to produce the yarns. By using needle looms[40] it has even become possible to manufacture felt-like textiles from these otherwise non-felting fibres. For the mechanical shrinkage of very fine fabrics, a newly developed open-width scouring machine with stock milling box has proved suitable[41]. Another very interesting machine for this purpose is likely to be the Compactor developed in the U.S.A.[42]. In this machine relaxation is achieved by compression forces acting in the direction of fabric delivery in such a way that not only weight but also bulkiness of the fabric increase. Chemical shrinking methods seem to be full of promise, although they have not yet been brought to technical maturity. Unfortunately, it is not yet possible to deal with this topical problem by mathematical principles, as computation must include not only fibre variables but also those of yarn and fabric. It seems, however, very probable that one day we may be able to calculate in advance precisely the data needed to produce a fabric with the desired crease-resistance, a property so important for the sales appeal of a fabric. All these problems, however grave, are only today's problems. When we have solved them, as I am quite sure that we shall do in due course, the gates to the future will not necessarily be open, and entirely different routes may have to be followed.

FUTURE OUTLOOK

When we look into the future we cannot talk in terms of finishing problems alone, but must widen our field of vision to survey the whole textile field, an approach well in line with the example set us by George Douglas.

At the present stage of textile technology, the textile industry is engaged in a fierce conflict between the traditional natural fibres and the progressive man-made fibres. Most of all the finishing problems in recent years were induced by this conflict, and once the problems were solved the conflict raged even fiercer than before.

We are, in fact, on the verge of a new epoch, which is attempting to do the work of Nature's innumerable small factories (as the animals or plants concerned may well be called) in large comprehensive units, to produce a fibre that is not only theoretically superior to the natural materials but has indeed been proved to be so. Increasing knowledge of body comfort shows with clarity that neither the natural raw material nor a highly developed synthetic fibre can guarantee the body comfort of a particular garment. Fabric properties such as construction and stability are largely superimposed on fibre properties. The protection we seek in clothing, the comfort we hope to experience, are not so much provided by the fibres as by air content and skin contact of the fabrics. The fur of animals, probably the ideal protection, rarely contains less than 90% by volume of air[43]. This air is responsible for heat exchange and moisture transport. In future developments of our textiles, therefore, we must aim at reproducing as closely as possible this skin–hair–air system of the animals by suitable construction of our textiles. This aim can no longer be attained by the textile finisher alone. It is a combined task for fibre supplier, spinner, weaver, dyer, and finisher.

No doubt, the more recent developments represent a step in this direction. I am thinking, for instance, of the methods for the manufacture of textured yarns, which have already found wide application here. With these yarns light and lofty fabrics may be produced. These fabrics represent a new era in textiles. As yet we are only at the beginning. For the time being the methods are limited to filament yarns. Blended yarns can, therefore, be produced only by combination of synthetic and regenerated cellulose filament yarns. Production methods are generally slow and, therefore, expensive. The new technique would be enhanced if fibre manufacturers were to develop special yarns for it, e.g. yarns with irregular cross-sections or of particularly low specific gravity.

Above all, however, efforts should be undertaken to equip spun yarns with the effects achieved in the texturising of filament yarns. Spun yarns are still ideal for the construction of a fabric, as they provide better irregular distribution and complete relaxation of the fibres necessary to achieve bulky, crease-resistant textiles. Only spun yarns allow blending of synthetic and natural fibres— the obvious way to bring together the two contestants in holy wedlock. First steps in this direction may be seen in the processes for the manufacture of tow-to-top and high-bulk yarns. Much ought still to be done in developing spinning techniques that are better suited to the synthetic fibres than are the traditional methods.

What we are really aiming at is the control of the third dimension in the fabric. Fabrics are normally sold by length and width, dimensions required by the making-up industry. The third dimension, viz. the thickness of the fabric, which may be the decisive factor for the comfort of the wearer, is expressed only vaguely as weight/yard. The vital factors, namely specific gravity and volume of air, are not taken into account. Not

until the third dimension of fabrics has been brought under firm control will the textile finisher make real progress in his tasks.

Fabrics of defined bulky structure can be finished not only by standard methods; but the textile finisher may also adjust air volume and skin contact of the fabrics to the necessities of body comfort, e.g. by controlled shrinkage without pressure. This would be a step nearer to overcoming another tremendous task in front of us, namely the realisation of comfort in clothing.

Complete knowledge of the inter-relationships between garment and human body should make it possible for us to provide the maximum of wear comfort for any type of person, any type of climate, and any type of use.

Unfortunately, however, the interrelations between the human body, because of its physiological and psychological reactions, and textiles, because of their multiplicity and variation in structure, are very complex[44,45]. An attempt has therefore been made to get a picture of the relationships involved by exploring the fundamental physico-chemical variables. These include—thermal insulation, moisture absorption and removal, air and gas permeability, light permeability, removal of skin grease, absorption and adsorption of odour, and the biological and biochemical effects of textiles on the skin.

In a series of investigations the required methods of measurement to determine these values, and the required conditions, have been worked out[46]. Although the resulting observations are extremely valuable, the problem has not been entirely solved. The basic variables—however clear they may appear—do not yet provide the key to the solution. They represent, unfortunately, no more than a simplification of the phenomena to be studied, so that in wearing tests contradictions between objective measurements and subjective statements by the wearer frequently occur[47].

As, however, the human body and textiles obey natural laws strictly, with certain variations, this problem, too, must be capable of solution if the experiments are arranged clearly and precisely. The correct measuring technique can, in this case too, be established with the aid of electronic computers, which check the mathematical rank correlation of the subjective findings and may reduce or even replace time-consuming wearing tests.

This brings me to the end of my lecture, which I hope has surveyed to some extent the wide field of textile finishing. Side by side with many successes, I have had to draw your attention to much that still awaits solution. It is my hope that you will find the gap closed if, in a few years' time, you were to choose tonight's subject for another George Douglas lecture.

* * *

May I thank the George Douglas Lecture Committee for their invitation to address you tonight. I have appreciated this as a great honour.

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Some Chemical Foundations of Fast Colour Printing

F. R. ALSBERG, W. CLARKE, and A. S. FERN

Meetings of the Northern Ireland Section held in Thompson's Restuarant, Belfast, on 14th January 1958, Mr. W. J. Macnab in the chair; and of the Manchester Section held at the Textile Institute, Manchester, on 17th January 1958, Mr. J. W. Reidy in the chair

The chemical foundations of the printing of cotton fabrics with vat, onium, and reactive dyes are discussed. Quantitative measurements of the intrinsic properties of vat dyes and of reducing systems enable a clear picture of the printing process to be obtained. The comparatively simple basis of fixation of onium dyes is discussed in relation to printing recipes and fixation conditions. Quantitative studies with reactive dyes show the effects of the solubilising agent, alkali, and fixation conditions on the percentage fixation obtainable under printing conditions.

I often say that if you can measure that of which you speak and can express it by a number you know something of your subject, but if you cannot measure it your knowledge is meagre and unsatisfactory.

Lord KELVIN

Introduction

The modern textile printer has been described as a "juggler with dyestuffs"; but just as a juggler has to exercise his skill in conformity with the laws of gravity and dynamics, so the textile printer must exercise his skill in conformity with fundamental chemical and physical laws.

Today the printworks chemist or colourist probably spends more effort in maintaining the volume and quality of production (in that order) than on discovering the reasons why certain effects do, and others do not, take place. The chemistry or physics of a process is often overlooked when applying dyes because of the necessity of producing a satisfactory result quickly and economically under works conditions. It is hoped that, by stating some of the chemical processes involved in fast colour printing, a stimulus will be provided for others to solve the many problems which must arise where several, usually conflicting, factors are simultaneously present.

"Fast colour printing" is defined as the process of producing on fabric a design which is fast enough to light, washing, rubbing, and other common agencies to last at least the life of the substrate on which it is printed. This definition is too academic for everyday use, and a more popular concept will be adopted— that fast colour printing entails the application of vat, soluble vat, azoic, onium, and reactive dyes, and excludes acid, basic, direct, and chrome dyes. Pigment printing compositions are omitted, not because they are incapable of giving "fast" effects, but partly because most printers use them in the knowledge that chemical control is not required to the same extent as for other dyes, and partly because the chemistry of the system does not concern the colouring matter so much as the resin binding the pigment to the fibre. It is proposed to consider only cellulosic fibres in this review.

Prints of high wet fastness on cellulose can be obtained by four basic methods—

(1) PIGMENT FIXATION— Originally, albumen was used to bind either naturally occurring or man-made inorganic pigments. Modern pigment printing systems take full advantage of the developments which have taken place in pigment and synthetic resin technologies [1].

(2) PRODUCTION IN THE FIBRE OF WATER-INSOLUBLE DYE AGGREGATES BY THE INTERACTION OF SMALL COMPONENTS— Examples are the formation of azoic pigments from coupling and diazo components, the oxidation of simple organic bases, and the formation of phthalocyanines by "polymerisation" of simple precursors in presence of a metal compound.

(3) PRODUCTION IN THE FIBRE OF WATER-INSOLUBLE DYE AGGREGATES DERIVED FROM A TEMPORARILY WATER-SOLUBLE PIGMENT DERIVATIVE— This type of printing is illustrated by vat dyes, which are converted to the leuco form by alkaline reduction, sulphuric esters of leuco vat dyes, and onium dyes.

(4) REACTION OF DYE WITH THE FIBRE SUBSTANCE— This is a recent development, in which a water-soluble azo, anthraquinone, or phthalocyanine reactive structure is condensed with the hydroxyl groups of cellulose.

In this paper, only vat, onium, and reactive dyes will be considered, since these three groups represent distinct stages in modern printing technology. Vat dyes have been available for many years, and a vast fund of empirical knowledge exists, only recently fortified by quantitative studies. Onium dyes have been marketed for about ten years, but in view of their inherently simpler application techniques there has been little incentive to commence fundamental work except when variations of printing recipes are involved. Finally, although the reactive dyes have been available for only two years, a considerable amount of fundamental knowledge has been gained. It is hoped that by simultaneously acquiring practical experience and quantitative information under controlled conditions, the printer of fast colour styles can be helped more effectively than when vat dyes were first made available.

I— Vat Dyes

In recent years a considerable amount of fundamental work has led to a better understanding of the chemical and physicochemical foundations of the vat-dye printing process. The present state of knowledge is summarised in Table I. The studies leading to this summary will now be briefly reviewed.

TABLE I

Inter-relation of Factors affecting Printing

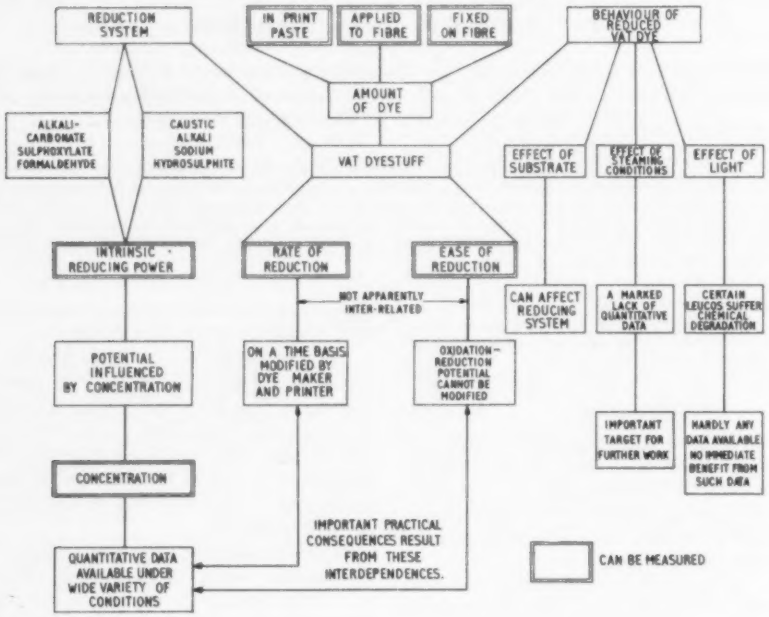

REDUCTION SYSTEM
IN PRINT PASTE
APPLIED TO FIBRE
FIXED ON FIBRE
BEHAVIOUR OF REDUCED VAT DYE
AMOUNT OF DYE
ALKALI-CARBONATE SULPHOXYLATE FORMALDEHYDE
CAUSTIC ALKALI SODIUM HYDROSULPHITE
VAT DYESTUFF
EFFECT OF SUBSTRATE
EFFECT OF STEAMING CONDITIONS
EFFECT OF LIGHT
INTRINSIC REDUCING POWER
RATE OF REDUCTION
EASE OF REDUCTION
NOT APPARENTLY INTER-RELATED
CAN AFFECT REDUCING SYSTEM
A MARKED LACK OF QUANTITATIVE DATA
CERTAIN LEUCOS SUFFER CHEMICAL DEGRADATION
POTENTIAL INFLUENCED BY CONCENTRATION
ON A TIME BASIS MODIFIED BY DYE MAKER AND PRINTER
OXIDATION-REDUCTION POTENTIAL CANNOT BE MODIFIED
IMPORTANT TARGET FOR FURTHER WORK
HARDLY ANY DATA AVAILABLE NO IMMEDIATE BENEFIT FROM SUCH DATA
CONCENTRATION
IMPORTANT PRACTICAL CONSEQUENCES RESULT FROM THESE INTERDEPENDENCES.
QUANTITATIVE DATA AVAILABLE UNDER WIDE VARIETY OF CONDITIONS
CAN BE MEASURED

TABLE II

System Reduction Potential (in British gum thickening)				Potential (mv.)	Vat Dye Leuco Potential	*Colour Index* Designation
K_2CO_3	NaOH	$NaHSO_2$, HCHO, $2H_2O$	$Na_2S_2O_4$			
				-700		
					-734	Durindone Printing Scarlet YS Paste (10%) — C.I. Vat Red 45
				-757	-753	Caledon Printing Yellow GNS Paste (10%) — C.I. Vat Yellow 1
					-760	Caledon Printing Red BNS Paste (10%) — C.I. Vat Red 35
				-800		
13·5	–	1	–		-817	Durindone Printing Brown GS Paste (10%) — C.I. Vat Brown 5
13·5	–	2	–		-845	Caledon Printing Orange 6RS Paste (10%) — C.I. Vat Orange 3
13·5	–	4	–		-877	Caledon Printing Jade Green XBNS Paste (5%) — C.I. Vat Green 1
				-896		
13·5	–	6	–	-910	-900 -901	Caledon Printing Purple 4RS Paste (5%) — C.I. Vat Violet 1
					-923	Caledon Brown RS Paste Fine (15%) — C.I. Vat Brown 3
13·5	–	8	–		-926	Caledon Printing Red 3B 200 Paste (7·5%) — C.I. Vat Red 10
				-950		
				-960	-958	Caledon Brilliant Violet RS Paste (10%) — C.I. Vat Violet 17
—	8	2	–	-980	-979	Caledon Printing Yellow 5GS Paste (10%) — C.I. Vat Yellow 2
13·5	–	–	8	-1000		
				-1025		
—	8	–	8	-1030		
				-1082		
—	8	8	–	-1100		

1. EASE OF REDUCTION

Measurements of the "leuco potential" by Linneken, Grand, and Fordemwalt [2], and by Marshall and Peters [3], were used to indicate the relative ease of reduction of a number of vat dyes under printing conditions. This potential is an inherent property of the dye. Dyes with a high leuco potential are usually difficult to print, especially if they are also slow to reduce.

Measurements of the potentials developed by a variety of reducing systems were also made by Marshall and Peters [4]. Table II has been constructed on the basis of their data. It shows the potentials developed by a range of sodium formaldehyde-sulphoxylate concentrations (in potassium carbonate) in relation to the leuco potentials of a number of vat dyes. If it is assumed that during steaming the alkali concentration remains substantially unaltered, while the concentration of sodium formaldehyde-sulphoxylate falls, Table II shows the level of reducing agent concentration at which a given vat dye fails to be reduced. The high potentials developed by replacing potassium carbonate by sodium hydroxide, and the potential developed by the sodium hydroxide–sodium hydrosulphite system, are noteworthy. Because some of these systems are unstable, special methods, e.g. "flash-ageing" (see below), have to be adopted to derive practical benefit from their strong reducing power.

2. RATE OF REDUCTION

There is no direct dependence of the rate of reduction of a vat dye on its ease of reduction. In vat printing, only a limited time is available for the dye to be reduced and absorbed, and a rapid rate of reduction is desirable. The rate can be increased by the presence of reduction catalysts and to a lesser extent by altering the physical form of the dye. Most dye manufacturers market special "Printing" brands of vat dyes for this reason. A method of measuring rates of reduction of vat printing pastes during steaming was devised by Marshall and Peters [4], who used the time for 50% of the maximum reduction as a convenient parameter. Times of 5–100 sec. were recorded for a range of eleven dyes, the majority of which were printing brands.

3. QUANTITATIVE MEASUREMENTS OF THE VAT PRINTING PROCESS

Provided that the amount of vat dye fixed under printing conditions can be measured, a general picture of its printing behaviour may be established. Michie and Thornton [5] carried out such experiments, determining the amount of dye fixed as a percentage of that originally applied. Variations in the concentration of sodium formaldehyde-sulphoxylate in the printing paste and in the steaming time were made. Vat dyes were divided into three broad categories—

(*a*) Dyes almost insensitive to alterations in the concentration of sodium formaldehyde-sulphoxylate in the printing paste, e.g. Caledon Printing Yellow GNS paste (C.I. Vat Yellow 1).

(*b*) Dyes which require a specific "threshold" concentration of sodium formaldehyde-sulphoxylate before satisfactory fixation can occur (the concentration may vary widely from dye to dye), e.g. Caledon Printing Jade Green XBNS paste (C.I. Vat Green 1).

(*c*) Dyes inherently unsuitable for application from print pastes containing potassium carbonate–sodium formaldehyde-sulphoxylate. Dyes in this group are hypersensitive to the concentration of reducing agent. Too little gives very low fixation, and an excess again lowers fixation on prolonged steaming, probably owing to destruction of dye, e.g. Caledon Brown RS paste fine (C.I. Vat Brown 3).

Table III shows the fixation and other characteristics of a dye from each of the above groups. It is constructed from data of Marshall and Peters [4] and Michie and Thornton [5].

4. DECOMPOSITION OF SODIUM FORMALDEHYDE-SULPHOXYLATE

(*a*) *Effect of the Substrate*

In general the substrate does not interfere with the fixation of a vat dye printed on it. If the

TABLE III

Fixation Characteristics of Vat Dyes

Dye and Print Paste Concentration	*Colour Index* Designation	Leuco Potential (mv.)	Time for 50% of Max. Reduction (sec.)	Time for 80% of Max. Fixation (sec.)	Fixation (%) Concentration of Sodium Formaldehyde-sulphoxylate in Print Paste								
					4%			8%			16%		
					Steaming Time (min.)			Steaming Time (min.)			Steaming Time (min.)		
					4	8	16	4	8	16	4	8	16
Caledon Printing Yellow GNS Paste (10%)	C.I. Vat Yellow 1	−753	5*	270	73	86	92	76	89	94	76	89	94§
Caledon Printing Yellow 5GS Paste (10%)	C.I. Vat Yellow 2	−979	45†	900	25	26	29	50	63	75	54	65	75‖
Caledon Brown RS Paste Fine (15%)	C.I. Vat Brown 3	−923	90‡	400	5	5	5	25	55	75	52	66	68¶

All printing pastes contained 13·5% K_2CO_3 and 5% glycerol, and were prepared from a British gum thickening.

* Measurement made on 1% print paste

† Print paste concn. 3·5%

‡ Print paste concn. 3·0%

§ Maximum fixation

‖ Maximum fixation achieved = 80% (steaming time 16 min., sodium formaldehyde-sulphoxylate concn. 10%

¶ Maximum fixation achieved = 87% (steaming time 16 min., sodium formaldehyde-sulphoxylate concn. 9·5%)

TABLE IV

Effect of Fabric on the Decomposition of Sodium Formaldehyde-sulphoxylate during Steaming

(Initial concn. 8% by wt. in print paste)

Time of Steaming (min.)	Concentration remaining after steaming on—				
	Mercerised Cotton Sateen	Viscose Rayon Staple	Cuprammonium Rayon	Linen	
				Caustic-shrunk (fully bleached)	Unbleached
1	5·9	5·9	5·8	6·2	5·2
3	4·0	3·6	3·7	3·7	3·0
5	2·5	2·5	2·2	2·3	1·8
8	1·7	1·5	1·1	1·3	0·6
11	1·1	1·0	0·5	0·6	0·1
15	0·7	0·6	0·3	0·3	0·1
20	0·3	0·5	0·3	0·3	—

TABLE V

Decomposition of Sodium Formaldehyde-sulphoxylate during Storage of the Print before Steaming

Dye in print paste ...	None		10% Caledon Printing Yellow GNS Paste (C.I. Vat Yellow 1)		10% Caledon Red 5GS Paste Fine (C.I. Vat Red 42)	
Leuco potential ...	—		−753 mv.		−900 mv.	
Temp. of drying ...	Room	70°c., then Room	Room	70°c., then Room	Room	70°c., then Room
Time of Storage (hr.)	Concn. of Sodium Formaldehyde-sulphoxylate remaining (initial concn. 8%)					
0	8	8	8	8	8	8
21	6·9	7·5	5·3	6·2	6·5	7·0
44	6·1	6·5	3·5	4·6	6·1	5·1
69	4·5	5·1	2·2	2·9	3·9	4·9

substrate contains substances capable of reacting with sodium formaldehyde-sulphoxylate, some loss of colour yield may occur unless excessive quantities of reducing agent are present. Linen fabrics before bleaching will behave in this way, and if a half-bleached linen is required, as in certain types of furnishings, scouring should be taken at least to the stage where undue loss of reducing agent during steaming does not occur.

Table IV, from the data of Thornton and Liquorice [5], shows how the concentration of sodium formaldehyde-sulphoxylate falls during steaming with a number of different fabrics. The horizontal lines indicate the approximate times after which the system fails to maintain an effective concentration of 1%. At this level only two of the eleven dyes listed in Table II are still capable of being reduced.

(b) Precautions to be observed in the Processing of Fabrics printed with Vat Dyes by the Sodium Formaldehyde-sulphoxylate Process

In their simplest form these precautions are—

(i) To ensure that a sufficient quantity of reducing agent is present to reduce any vat dye printed in the design and maintain it in the reduced, soluble form until the maximum amount has been fixed under a given set of conditions. It has already been shown that the concentration of reducing agent necessary varies from dye to dye, and that variation of the alkali can influence the power of the reduction system.

(ii) To avoid premature loss of reducing agent before or during steaming.

The following factors have to be taken into account—

LOSS OF SODIUM FORMALDEHYDE-SULPHOXYLATE DURING STORAGE OF THE PRINTS BEFORE STEAMING—This is accelerated by drying slowly, which results in the prints having an appreciable moisture content for a long period. The presence of an easily reduced vat dye of low leuco potential increases such losses, whereas a difficultly reduced vat dye has very much less effect under the same conditions [6]. This is shown in Table V.

DECOMPOSITION OF SODIUM FORMALDEHYDE-SULPHOXYLATE DURING STEAMING— *Effect of initial concentration* It has been shown that the rate of decomposition of sodium formaldehyde-sulphoxylate is increased if the initial concentration is low [6].

TABLE VI

Rate of Decomposition of Sodium Formaldehyde-sulphoxylate

Initial Concentration (% in print paste)	Time for 50% Loss (min.)
4	1·5
8	2·8
12	5·0

The times for a 50% loss during steaming at three different concentrations are given in Table VI. It is therefore false economy to lower the concentration of reducing agent unduly. With concentrations as high as 12%, safe working conditions would arise provided that prints entered the steamer with this concentration. Even after 5 min. steaming it is clear from Table IV that considerable reducing power remains.

Variation in the alkali used Two effects have been investigated in some detail— the replacement of potassium carbonate by sodium carbonate and

by caustic soda in the printing pastes. Sodium carbonate, being efflorescent, has an advantage where drying conditions are poor and is to some extent an insurance against this type of loss of reducing agent. Caustic soda additions increase the reducing power of the system, but unfortunately also reduce its stability. While it is true that judicious additions of caustic soda allow certain dyes to be printed that otherwise would fail to reduce and fix properly, the inherent instability will, unless great care is taken, result in premature loss of reducing agent before steaming. Table VII shows that there is no significant difference between potassium carbonate and sodium carbonate in respect of their effect on the loss of reducing agent. With caustic soda, however, the reducing agent decomposes at a much more rapid rate, and the drying operation prior to steaming results in the loss of almost one-quarter of the sodium formaldehyde-sulphoxylate.

TABLE VII

Effect of Various Alkalis in the Print Paste on the Rate of Decomposition of Sodium Formaldehyde-sulphoxylate during Steaming

(Initial concn. of reducing agent 8% in print paste containing alkalis)

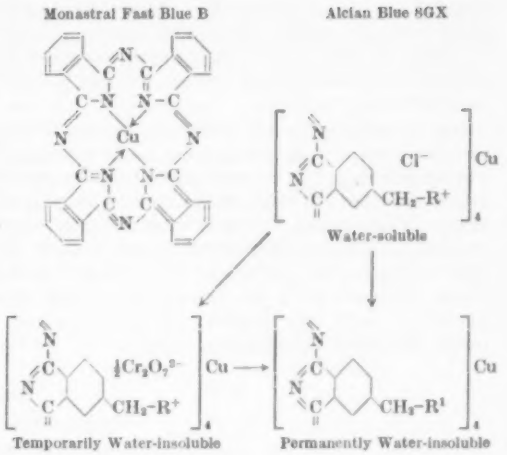
Time of Steaming (min.)	Concentration of Reducing Agent remaining		
	13·5% K_2CO_3 (anhyd.)	6% Na_2CO_3 (anhyd.)	6% NaOH (flake)
0	8	8	6·2
1	5·8	6·2	3·7
3	4·3	5·1	3·0
5	3·4	3·3	2·2
8	2·4	2·4	1·8
11	1·9	1·8	1·4
15	1·3	1·1	1·1
20	0·9	0·8	0·8

II— Onium Dyes with temporarily Solubilising Groups

The first onium dye, Alcian Blue 8GS (C.I. Ingrain Blue 1), was marketed in December 1947. Today there are seven dyes in the Alcian X series— four blues, two greens, and a yellow. There are few countries in the world where these dyes have not been used for printing. This is attributed to two main factors— (*a*) the need for a bright, fast turquoise blue on cotton, and (*b*) the simplicity and reliability of the Alcian system under a wide variety of operating conditions.

In contrast to the vat dyes, very few quantitative data on the fixation of Alcian dyes have been published. This present discussion will unfortunately do little to remedy this, although it is hoped that a summary of the basic essentials of application techniques will assist those who are already experienced in printing onium dyes, as well as stimulate those who are not.

Alcian Blue 8G was the outcome of a number of efforts to solubilise copper phthalocyanine and obtain a dye which could be printed in fast styles. The technique is one of temporary solubilisation based on a two-stage reaction. The main reactions which occur during printing with Alcian Blue 8GX are—



Fixation can be effected by two methods—

(1) When printed from a system containing a volatile acid and an alkaline salt (e.g. acetic acid and sodium acetate), the permanently water-insoluble form is produced directly by heating or steaming: loss of acid leads to the alkaline conditions required for insolubilisation.

(2) When the dye is steamed in presence of a non-volatile acid and a solvent, some dye is present in the water-soluble form after steaming, and this must be immobilised by complex formation with e.g. dichromate anions. Further treatment in hot *alkaline* soap or detergent solution leads to formation of the permanently water-insoluble product.

The presence of cationic solubilising groups has the following effects on the properties of Alcian dyes—

(i) Alcians form precipitates with anionic dyes (i.e. the majority of water-soluble dyes) and cannot be used in admixture with them. Anionic wetting agents and other printing auxiliaries cannot be used for the same reason.

(ii) The Alcian solubilising groups are adversely affected by strong alkalis and by reducing and oxidising agents. Contamination of the printing paste with other than traces of these substances must therefore be avoided.

PRINTING RECIPES

In the course of the investigations that led to the establishment of the Alcian recipes, the following facts emerged—

(1) The rate of insolubilisation varies from dye to dye, being slowest with Alcian Blues 8GX and 7GX and most rapid with Alcian Yellow GX (C.I. Ingrain Yellow 1).

(2) The best build-up, brightness, and rubbing fastness are obtained by allowing the dye to penetrate into the fibre and restraining insolubilisation. This requires a relatively high concentration of a solvent for the dye as well as a non-volatile acidic component. This technique is the basis of the Alcian Developer X recipes.

(3) Where a printing style requires steaming to be minimised or avoided, insolubilisation may be

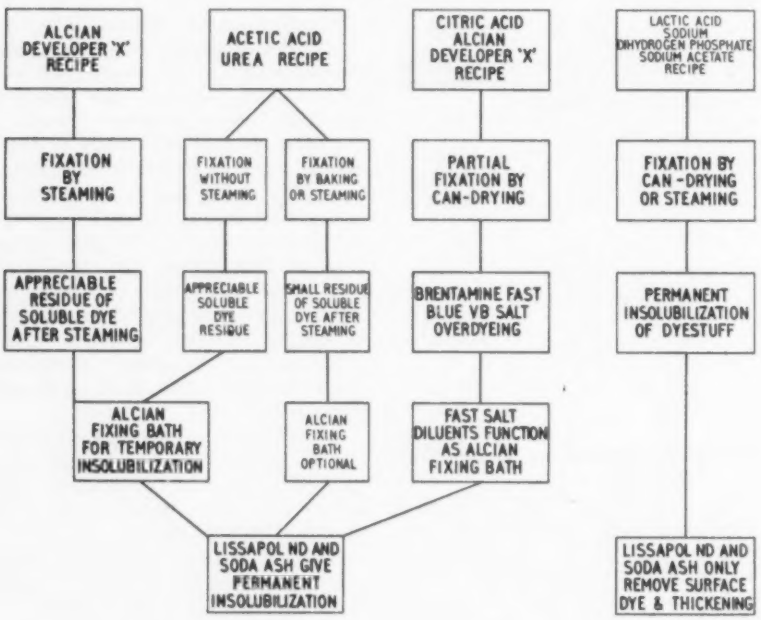
speeded up by replacing the non-volatile acidic component by acetic acid. This results in some lowering of yield and rubbing fastness, but for reasons of economy or expediency it is often acceptable in practice.

(4) Insolubilisation is most rapid when sodium acetate is present in the printing paste (or formed during printing, e.g. when Brenthol(ICI)-prepared fabrics are printed with Alcians containing acetic acid). This rapid insolubilisation limits such recipes to medium depths of Alcian Yellow GX and Alcian Greens 2GX and 3BX. Alcian Blues 2GX and 5GX are unsuitable for this type of recipe, since very marked dulling occurs as a result of surface precipitation of dye.

TABLE VIII

Printing Recipes for Alcian X Dyes

General properties


ALCIAN DEVELOPER 'X' RECIPE
ACETIC ACID UREA RECIPE
CITRIC ACID ALCIAN DEVELOPER 'X' RECIPE
LACTIC ACID SODIUM DIHYDROGEN PHOSPHATE SODIUM ACETATE RECIPE
FIXATION BY STEAMING
FIXATION WITHOUT STEAMING
FIXATION BY BAKING OR STEAMING
PARTIAL FIXATION BY CAN-DRYING
FIXATION BY CAN-DRYING OR STEAMING
APPRECIABLE RESIDUE OF SOLUBLE DYE AFTER STEAMING
APPRECIABLE SOLUBLE DYE RESIDUE
SMALL RESIDUE OF SOLUBLE DYE AFTER STEAMING
BRENTAMINE FAST BLUE VB SALT OVERDYEING
PERMANENT INSOLUBILIZATION OF DYESTUFF
ALCIAN FIXING BATH FOR TEMPORARY INSOLUBILIZATION
ALCIAN FIXING BATH OPTIONAL
FAST SALT DILUENTS FUNCTION AS ALCIAN FIXING BATH
LISSAPOL ND AND SODA ASH GIVE PERMANENT INSOLUBILIZATION
LISSAPOL ND AND SODA ASH ONLY REMOVE SURFACE DYE & THICKENING

A summary of the main features of the recommended recipes is given in Table VIII.

BULK PRINTING WITH ALCIAN DYES

The Alcian printing system has few variables which cannot be readily controlled. The components of the printing recipes do not lose their efficiency if the fabric is stored prior to steaming, within wide limits steam quality is not critical, and contamination of the steam with air has no adverse effect. With those recipes which require a fixing bath, however, this aftertreatment stage must be controlled if prints of the highest quality are to be obtained consistently.

Where only partial insolubilisation has occurred after steaming, that proportion of the Alcian dye which is still water-soluble must first form an insoluble salt with either the dichromate or the Azoguard (ICI) anion. The amount of sodium dichromate required to insolubilise Alcian Blue 8GX in its water-soluble form was determined as follows—

Plain-weave unmercerised cotton fabric was slop-padded (at 20°c.) with a solution containing, by weight, 5% Alcian Blue 8GX 300, 5% lactic acid (80%), and 5% Glydote B (ICI). The pick-up was 70% on the dry weight of cloth. The material was dried, and steamed for various times. Portions of equal area were then immersed for 5 min. in separate Alcian fixing baths at 20°c. containing 0·2% (wt./vol.) sodium dichromate and 0·2% (vol./vol.) acetic acid (glacial). Aliquots of the used fixing baths were acidified with hydrochloric acid, 1 g. of potassium iodide was added, and the residual dichromate was titrated with 0·1 N. sodium thiosulphate solution using soluble starch as indicator. Dye on the padded fabric was determined by extracting a known area repeatedly with lactic acid (50%) at 80°c., and estimating the dye colorimetrically in the usual way. A typical set of results is given in Table IX.

TABLE IX

Sodium Dichromate required to insolubilise Alcian Blue 8GX 300

Steaming Time (min.)	$Na_2Cr_2O_7.2H_2O$ (g./g. dye)
0	0·57
10	0·37
20	0·34
30	0·24

By taking 0·4 g. sodium dichromate per gram of Alcian Blue 8GX 300 as representing the amount

of water-soluble dye left under typical practical conditions, the rate of addition of dichromate can easily be calculated, taking into account such typical variables as—(*a*) strength of dye in the original print paste, (*b*) uptake of paste per unit area of cloth (proportional to depth of engraving and percentage coverage of the pattern), and (*c*) speed of the fabric through the fixing bath. For example, the following relation exists—

Print paste applied per 100 sq. metres of cloth, kg.	5
Dye concentration in print paste, g./kg.	10
Soaper speed, metres/min.	50
Sodium dichromate required, kg./hr.	0·6

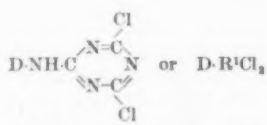
In view of the relatively small amounts of sodium dichromate involved it is false economy to use too little. In vat printing, even if the concentration of dichromate is too low, there is always the possibility of air oxidation preventing the vat dye from bleeding and staining the whites. It is most important to realise that with Alcian dyes a quite different chemical reaction is utilised, that air oxidation has no effect on the insolubilisation of Alcian X dyes, and that prolonged washing with cold water hydrolyses the Alcian–dichromate complex, liberating an Alcian derivative which can cause staining.

III—The Fixation of Reactive Dyes printed on Cotton

1. INTRODUCTION

The first three reactive dyes—Procion Yellow RS, Procion Brilliant Red 2BS, and Procion Blue 3GS (ICI)—were introduced early in 1956. The general printing properties of this short range were described by Vickerstaff [8]. The most important application method entails the use of a print paste containing dye, urea as solvent, sodium bicarbonate as acid-binding agent, and a sodium alginate thickening: fixation is by steaming, and a thorough washing off completes the process.

These first Procion dyes contain the dichloro-*s*-triazinyl reactive system, and may be written thus—



This is a highly reactive system, and with certain dyes maximum fixation takes place when the printed cloth is merely dried at 60°c. The reactions which occur during fixation may be represented as—

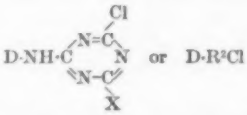
$$D\cdot R^1Cl_2 + Cell\text{–}OH \longrightarrow Cell\text{–}O\cdot R^1DCl + HCl \quad (i)$$

$$D\cdot R^1Cl_2 + 2H_2O \longrightarrow D\cdot R^1(OH)_2 + 2HCl \quad (ii)$$

The species $D\cdot R^1(OH)_2$ is a highly coloured, water-soluble, non-reactive dye usually possessing negligible substantivity for cellulose, and is formed to an extent depending on the dye, the fibre, and, to a lesser degree, the application conditions.

This reaction between dye and fibre has many desirable properties for the printer: it is almost insensitive to steaming time, temperature, and humidity, to the presence of air during fixation, and to storage of the print before and after fixation. On the other hand, exposure to acid before or during fixation results in partial neutralisation of sodium bicarbonate and can affect fixation. Furthermore, with such a reactive system, reaction (ii) can take place in the print paste at room temperature, leading to loss of colour value.

Printers in certain markets demand the highest possible print paste stability, and the development of Procion H dyes soon followed. These generally contain the monochloro-*s*-triazinyl reactive system—



Reactions similar to (i) and (ii) for the dichloro-*s*-triazinyl system occur with cellulose and water respectively, but in this case very little activity is apparent below about 100°c. Such dyes are therefore unsuitable for low-temperature fixation methods, but conversely possess very high print-paste stability at room temperature. The first member of this later series, Procion Brilliant Red H3BS, was followed by a wide range of Procion H dyes possessing similar characteristics.

Fortunately, the introduction of these less reactive dyes did not necessitate a major change in methods of application, for, provided that an adequate steaming treatment was given, they gave their optimum fixation from a print paste containing urea, sodium bicarbonate, and sodium alginate.

Bulk printing with Procion and Procion H dyes has confirmed that this cellulose–dye system is unaffected by most printworks variables, many of which would render vat dyes, for example, valueless. Those problems which have been encountered arose from unpredictable contamination by acidic foreign matter introduced by the substrate, during steaming, or, in screen printing, by the table adhesive. It was therefore considered desirable to study quantitatively some of the fundamental properties of the Procion–cotton system to obtain a better understanding of those factors which determine the success of the reaction.

2. EXPERIMENTAL

(*a*) *Materials*

Dyes and Auxiliary Products—Commercial quality.

Sodium Bicarbonate and Sulphuric Acid—A.R. quality.

Sodium Carbonate and Caustic Soda—A.R. quality.

Thickening—Based on 5% (wt./vol.) aqueous solution of Manutex RS (Alginate Industries Ltd.) containing 1·25% (wt./vol.) sodium hexametaphosphate.

Fabric—Plain-weave bleached unmercerised cotton, 60 picks, 76 ends/in., 5·3 oz./sq.yd., cuprammonium fluidity 5·2. Scoured before use in boiling water (twice) and cold water to remove traces of acid or alkali.

(b) *Printing*

Prints were produced on a laboratory hand-operated chromium-plated copper roller machine, with a $\frac{3}{4}$ in. stripe engraving 0·01 in. deep. Drying, steaming (6 min.), and washing-off processes were rigorously standardised.

(c) *Quantitative Estimations of Fixation*

Fixation was estimated by dissolving the print in concentrated sulphuric acid by the general method described for vat dyes by Michie and Thornton [5]. The solution was drowned in Dispersol VL (ICI) solution so that the final solution contained 10% (by vol.) H_2SO_4 and 0·2% (by vol.) Dispersol VL. Optical densities were measured on a Unicam SP 600 (Cambridge) spectrophotometer.

(d) *pH Measurements*

The print, 10 in. long and $\frac{3}{4}$ in. wide, was immersed in 100 ml. distilled water at room temperature for 1 hr. with occasional stirring. The alkalinity of the extract was measured with a pH-meter.

3. RESULTS AND DISCUSSION

(a) *Effect of Urea Concentration*

It is reasonable to assume that reaction with the cotton fibre can occur only in those areas to which the dye can diffuse in a water-soluble state. A solvent like urea might therefore be expected to assist in fixing heavy depths of those members of the Procion and Procion H ranges which possess comparatively low solubility in water. Fig. 1 and 2 show the effect of urea concentration on the fixation of Procion Yellow RS and Procion Brilliant Red 2BS, dyes of respectively low and high aqueous solubility.

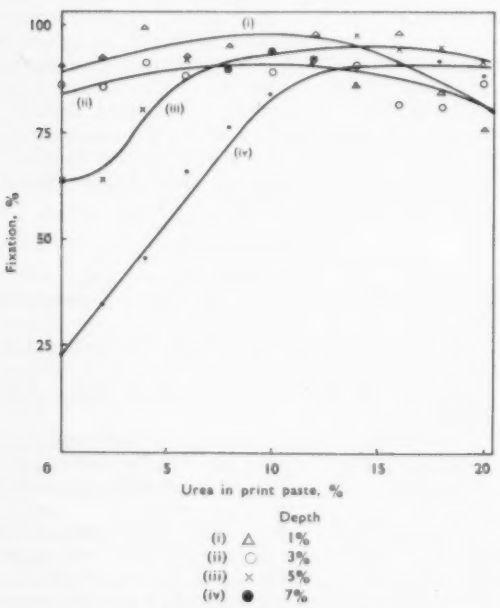


FIG. 1—Procion Yellow RS

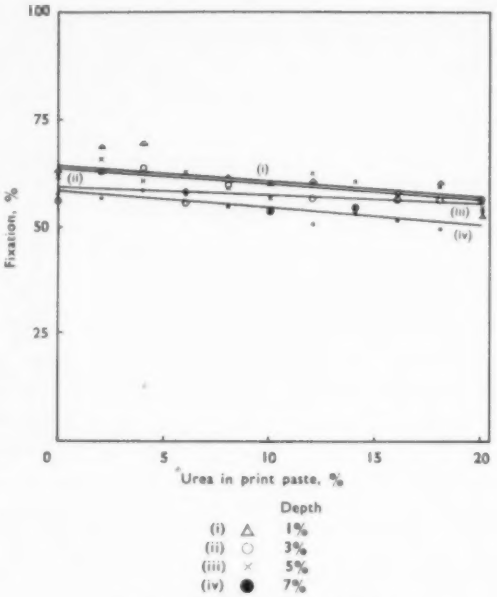


FIG. 2—Procion Brilliant Red 2BS

With Procion Brilliant Red 2BS, urea concentration is not critical and, in fact, increasing the amount of urea lowers the fixation slightly, presumably because of the greater tendency for dye to remain in the aqueous phase and react with water. Such a dye could be printed in absence of urea. With Procion Yellow RS, 10% urea is required at 1% and 3% depths, and the fixation does not fall off much on either side of this value. At greater depths at least 15% urea is necessary; below this value the dye is largely out of solution, and fixation drops very rapidly.

(b) *Effect of Sodium Bicarbonate Concentration*

Variation in fixation with bicarbonate concentration was determined using the optimum urea concentration found under (*a*). Fig. 3 and 4 show this relation for dyes differing widely in behaviour. Procion Yellow RS is typical of the more reactive dichloro-*s*-triazinyl type in that there is an optimum concentration of sodium bicarbonate, which increases as the dye concentration increases. In this case a deficiency of bicarbonate is more serious than an excess. With Procion Brilliant Red H3BS, the fixation increases steadily to a maximum, and does not fall significantly after this is reached.

From the composition of each dye can be calculated the amount of sodium bicarbonate theoretically required to neutralise all the hydrochloric acid formed during reaction with cotton or water. This amount and the optimum amount of bicarbonate (from Fig. 3) are plotted against depth applied for Procion Yellow RS in Fig. 5. The parallel relation between theoretical and optimum bicarbonate concentration suggests that this reactive system requires not only sufficient bicarbonate to neutralise the hydrochloric acid (this will depend on depth of dye applied) but also an additional constant quantity. It is suggested that

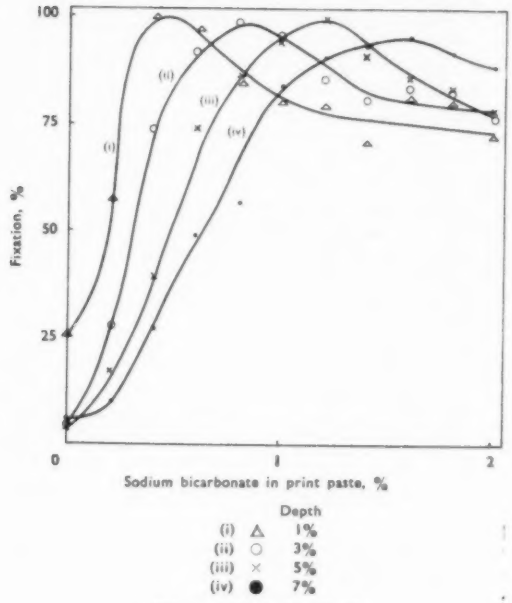


FIG. 3—Procion Yellow RS

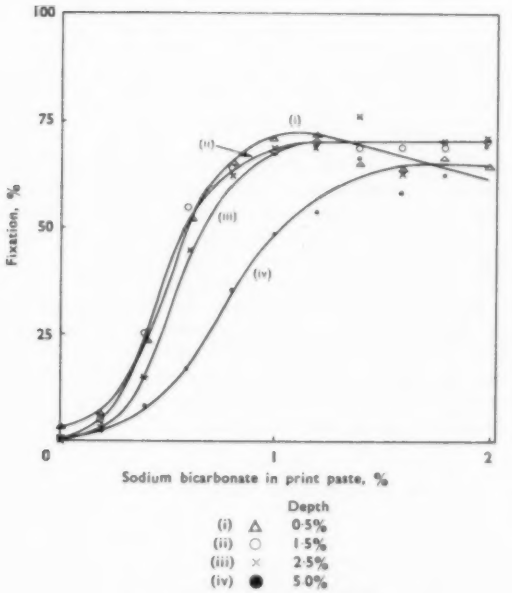


FIG. 4—Procion Brilliant Red H3BS

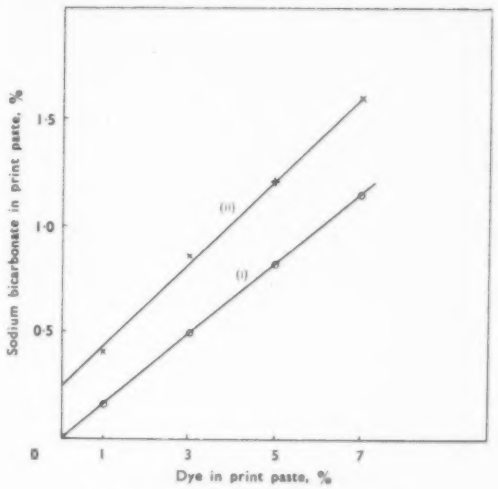

Sodium bicarbonate in print paste, %
Dye in print paste, %
(i) ○ NaHCO3 neutralised by HCl formed
(ii) × Optimum NaHCO3 in print paste

FIG. 5—Procion Yellow RS

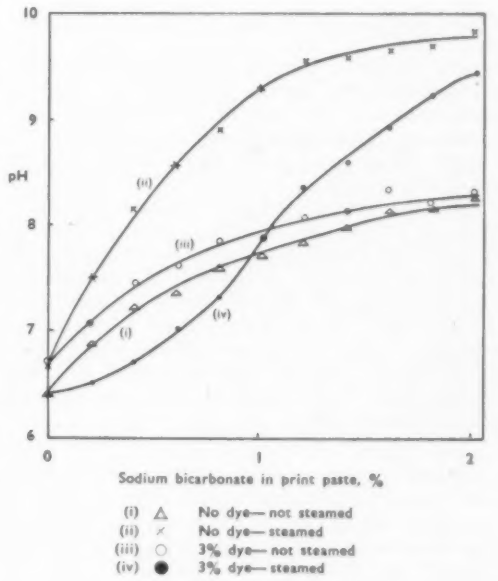


FIG. 6—Procion Yellow RS

this second amount is required either to bring the cellulose to the required degree of ionisation for maximum reaction to occur or to establish conditions in the aqueous phase where the reaction with water is at a minimum. With the less reactive Procion Brilliant Red H3BS, the optimum bicarbonate concentration is higher, either because a greater degree of ionisation of the cellulose is necessary, or because the rate of hydrolysis of this dye is favoured by lower pH conditions. Further work will be needed to elucidate this point.

The actual alkalinity of the printed fabric was studied for the same two dyes and for a print paste containing no dye: the results obtained from pH measurements of an aqueous extract are shown in Fig. 6 and 7. With no dye, the pH of an unsteamed print rises from 6·5 to 8·0 as the bicarbonate concentration rises from 0 to 2%. The presence of 3% Procion Yellow RS or of 2·5% Procion Brilliant Red H3BS does not greatly alter this behaviour. On steaming, in absence of dye, the pH rises rapidly to 9·5 and thereafter remains constant; this is due to conversion of bicarbonate to carbonate. With Procion Yellow RS present, some of the alkali is neutralised as a result of reaction with the

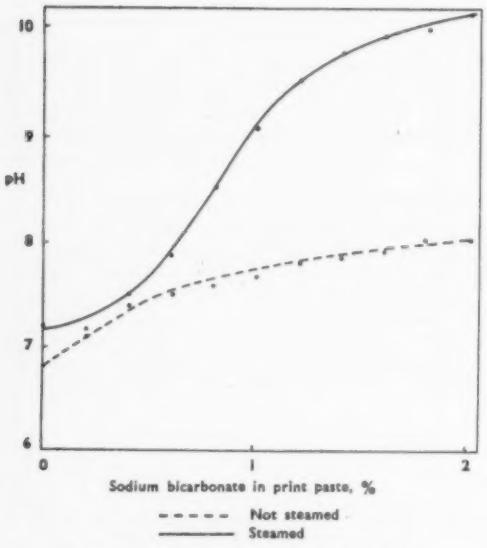


FIG. 7—2·5% Procion Brilliant Red H3BS

cotton, and the pH of the extract is accordingly lower than for the uncoloured paste. With Procion Brilliant Red H3BS similar behaviour is observed, although the pH rises to a slightly greater value at the maximum bicarbonate concentration. This difference could be attributed to a slight difference in the buffering action of these two dye preparations.

From the fixation and pH data in Fig. 3 and 6 (Procion Yellow RS) and Fig. 4 and 7 (Procion Brilliant Red H3BS) it is possible to deduce the pH of optimum fixation. It is essential to correct the aqueous-extract pH for the higher concentration of alkali which exists on the printed fabric during steaming; a separate experiment showed that under the conditions employed, cotton printed with this Procion paste is associated with 20% (on its own dry weight) of water.

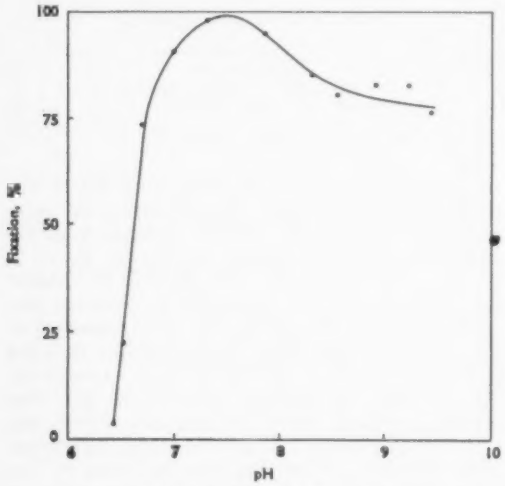


FIG. 8—3% Procion Yellow RS

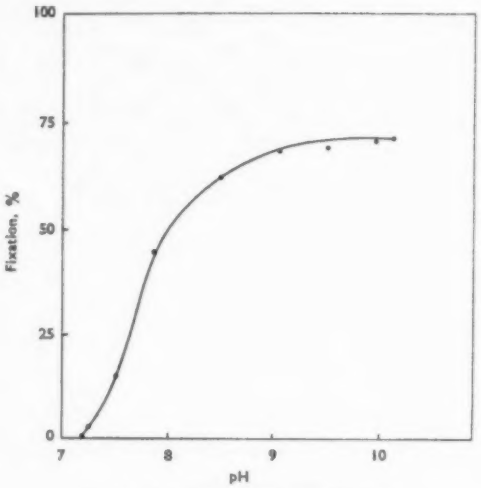


FIG. 9—2·5% Procion Brilliant Red H3BS

Fig. 8 and 9 show the optimum pH to be as indicated in Table X.

TABLE X

	Optimum pH Based on Extract	Optimum pH Corrected for Print	Optimum Fixation (%)
3% Procion Yellow RS	7·6	9·0	98
2·5% Procion Brilliant Red H3BS	9·25	11·25	70

The pH correction is open to criticism in that no allowance can be made for the temperature (100°C.) at which much of the fixation must occur. Nevertheless, these figures give some guide to the alkalinity required, and show adequately the difference between these two dyes.

The influence on fixation of an appreciably greater alkalinity in the print paste was studied for 2·5% Procion Brilliant Red H3BS by adding caustic soda to pastes originally containing either sodium bicarbonate or sodium carbonate. The results are shown in Table XI.

TABLE XI

Alkali Content of Paste (% by wt.) $NaHCO_3$	Na_2CO_3	NaOH	Fixation (%)
1·5*	—	—	66·0
1·5	—	0·01	66·5
1·5	—	0·03	65·5
1·5	—	0·10	67·0
—	0·95*	—	66·0
—	0·95	0·01	67·5
—	0·95	0·03	68·0
—	0·95	0·10	67·5

* These are equimolecular quantities.

It is concluded that additions of caustic soda up to 0·1% do not result in improved fixation of Procion Brilliant Red H3BS on unmercerised cotton.

(c) *Effect of Concentration of Water*

Owing to the formation of a useless by-product from these reactive dyes by hydrolysis, it is natural

to ask what happens if the amount of water in the system is reduced. It is unlikely that the cellulose reaction can occur at all in complete absence of a solvent for the dye, although many highly reactive dyes appear to react during a baking treatment. In fact, they have either reacted normally while the fabric is being dried after printing, or have been fixed in presence of molten urea, which acts as the solvent. A simple calculation shows that 1 g. Procion Brilliant Red 2BS requires only 0·02 g. water for complete hydrolysis (and hence 0·0067 g. water if two-thirds of the dye reacts with cellulose). In a typical print paste there will be about 80 g. water per 100 g. paste, and on the fabric during steaming about one-fifth of this, viz. 16 g. water. At worst 800 times as much water as that required to hydrolyse the dye will be present; therefore the use of printing systems containing as little as 5% or 10% of water should, in theory, offer no advantage over the conventional sodium alginate paste.

TABLE XII

System	Water Content (%)	Fixation (%)
Sodium alginate thickening	87	74
Water-in-oil emulsion	70	63
Oil-in-water emulsion	29	67

In fact, the fixation results in Table XII were obtained for 3% Procion Brilliant Red 2BS applied from print pastes containing different amounts of water, and fixed by a drying treatment.

(d) *Effect of Time of Steaming*

Fig. 10 shows the influence of steaming time on fixation for two dyes, each being printed at its optimum urea and sodium bicarbonate concentrations. 3% Procion Brilliant Blue RS fixes to about 20% without steaming, whereas Procion Brilliant Red H3BS shows negligible fixation under such conditions. Less than 5 sec. steaming is required to achieve the maximum fixation (65%) of Procion Brilliant Blue RS, and longer treatments lower the fixation slightly, owing to hydrolysis of the dye–fibre linkage or to dye decomposition. Procion Brilliant Red H3BS gives its maximum fixation (70%) only after 5 min. steaming. Prolonged treatment does not affect the amount fixed, but lower times must be avoided (e.g. the fixation after 1 min. is only 30%). Many of the later Procion H dyes possess even lower reactivity than Procion Brilliant Red H3BS, and must be steamed for at least 10 min. for optimum fixation.

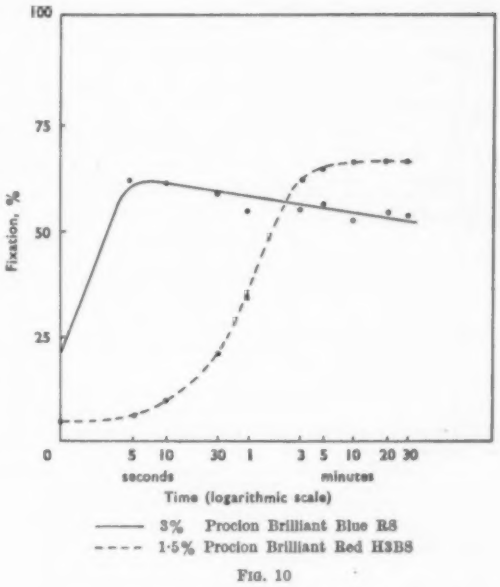


FIG. 10

(e) *Summary of Optimum Conditions*

Quantitative data of the type previously described now exist on five dyes, whose behaviour is summarised in Table XIII.

(f) *Fixation by Dry-heat Treatment*

Steaming can be dispensed with for the dichloro-*s*-triazinyl dyes, since they can be fixed equally well by a short dry-heat treatment (e.g. on steam-heated drying cans). The alkali required for fixation can be applied either as sodium bicarbonate in the print paste, or after printing and drying, by nip-padding the cloth in sodium carbonate solution before redrying. Fig. 11 shows the fixation of 3% Procion Brilliant Blue RS under such conditions, the degree of fixation with a steaming process being indicated at the left of the diagram. The lower reactivity of the Procion H group is again exemplified by 1·5% Procion Brilliant Red H3BS, whose behaviour under comparable conditions is shown in Fig. 12. In this case, can-drying is much less effective than steaming, and dyes of this type are not recommended for this type of dry-heat fixation.

In order to study more precisely the lowest temperature at which significant fixation of the more highly reactive Procion dyes can occur, prints of Procion Brilliant Red 2BS and Procion Brilliant Red H3BS containing sodium bicarbonate were stored for relatively long periods at temperatures

TABLE XIII

Summary of Optimum Conditions

Printing strength ...	1%			3%	5%		
Dye	Urea (%)	$NaHCO_3$ (%)	Fixation (%)	Time of Max. Fixation	Urea (%)	$NaHCO_3$ (%)	Fixation (%)
Procion Yellow RS	3	0·4	98	1 min.	13	1·2	97
Procion Brilliant Red 2BS	0	0·5	67	<5 sec.	0	0·8	67
Procion Brilliant Blue RS	5	0·5	76	<5 sec.	15	1·0	60
Procion Brilliant Red H3BS	0	1·0	72	5 min.	5	1·2	70
Procion Blue HBS	5	1·1	68	7·5 min.	9	1·5	65

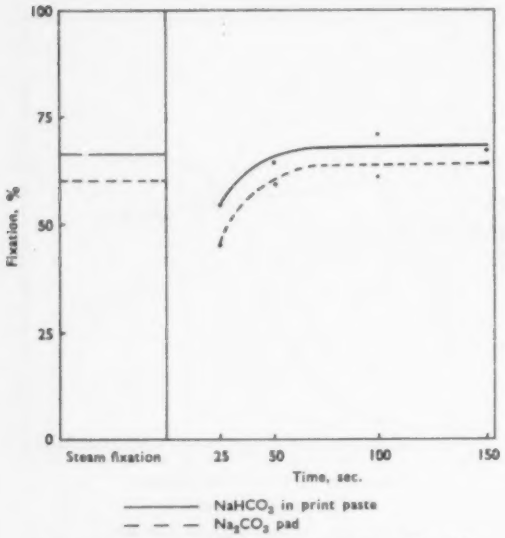


FIG. 11—Can Fixation of 3% Procion Brilliant Blue RS

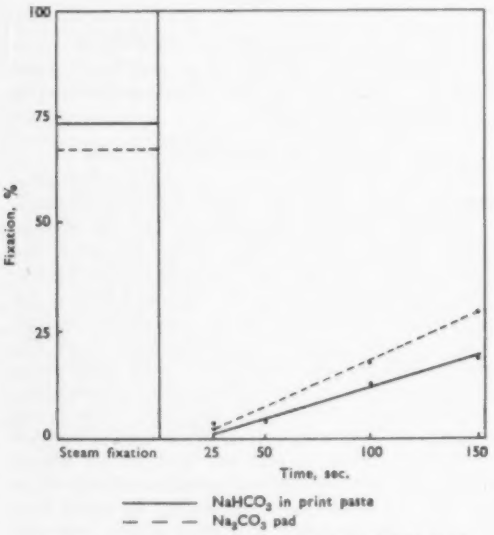


FIG. 12—Can Fixation of 1·5% Procion Brilliant Red H3BS

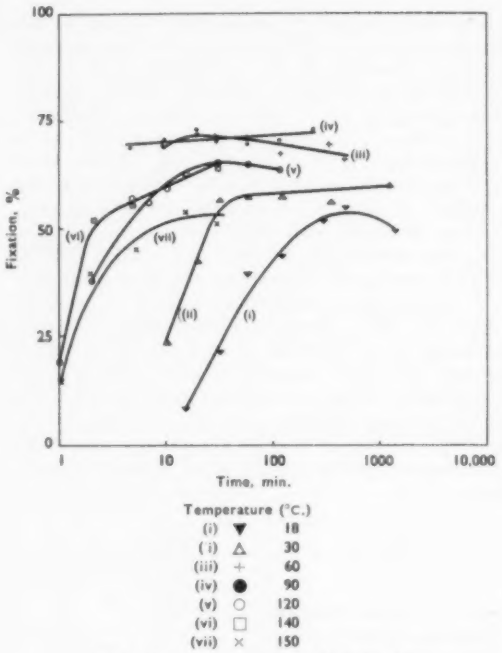


FIG. 13—4% Procion Brilliant Red 2BS

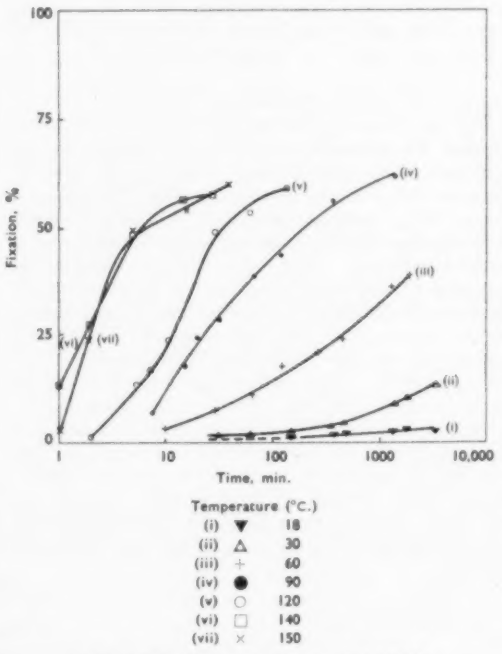


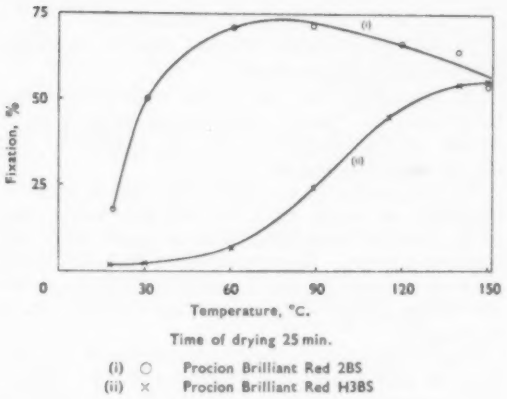
FIG. 14—2·5% Procion Brilliant Red H3BS

varying from 20°C. to 150°C., before being washed off. Fig. 13 and 14 indicate the rate of fixation at each temperature, and provide the data for Fig. 15, where the fixation occurring in 25 min. is plotted against temperature. Whereas Procion Brilliant Red 2BS gives optimum colour yield at about 65°C., Procion Brilliant Red H3BS does not fix efficiently even at 150°C. under these dry-heat conditions.

4. CONCLUSIONS

(1) Existing reactive dyes generally contain either a highly reactive dichloro-*s*-triazinyl system (Procion) or a less reactive monochloro-*s*-triazinyl system (Procion H). The proportion of urea required for optimum fixation on cotton depends on the solubility of the dye in water; the amount of sodium bicarbonate depends on the depth of dye printed, and is generally higher for Procion H dyes than for the more reactive Procions.

(2) The maximum fixation on cotton is not necessarily determined by the reactive system



Time of drying 25 min.

(i) ○ Procion Brilliant Red 2BS
(ii) × Procion Brilliant Red H3BS

FIG. 15

which the dye contains, but may be influenced also by the solubility of the dye, its rate of diffusion, and its affinity for cellulose. It is almost independent of dye concentration in the range 1–5%.

(3) Those dichloro-*s*-triazinyl dyes so far examined fix to the maximum extent in less than 1 min. steaming, and certain members of this group require as little as 5 sec. The monochloro-*s*-triazinyl dyes require at least 5 min. steaming. Only the former group can be fixed appreciably when the temperature is lower than 100°C., or when a dry-heat fixation on steam-heated cans is given.

* * *

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Some Experiments on the Effect of Dye, Fibre, and Atmosphere on Light Fastness

G. SCHWEN and G. SCHMIDT

Meeting of the Scottish Section held at the St. Enoch Hotel, Glasgow, on 24th September 1957, Mr. W. G. B. Grant in the chair

The influence of light on dyeings on various fibres of basic dyes of different structures has been investigated in dry and moist oxygen and nitrogen. The results have been compared with those obtained under similar conditions in a normal atmosphere. It has been found that some dyes fade more rapidly in oxygen than in nitrogen. Contrary to expectation, certain dyes were found to fade much more rapidly in the nitrogen atmosphere than in the oxygen atmosphere or under normal atmospheric conditions. As expected, all the dyeings were found to fade more rapidly in a moist atmosphere. An attempt has been made to interpret the results as either an oxidation or a reduction of the dye, depending on its constitution. The water contained in the fibre is regarded as an essential component in the oxidation–reduction process.

1. INTRODUCTION

In the light of present knowledge it is now quite clear that the system comprising dye and fibre together— or, rather, dye, fibre, and further components— is necessary to define the light fastness of a particular dyeing. One cannot quote the light fastness of a dye alone.

The earliest observations on the variation of light fastness of a dye on different fibres were made on Indigo (C.I. Vat Blue 1), which is fast to light on wool (grade 7–8) but not so fast on cotton (grade 3). Furthermore, various workers in this field have found that a dyeing fades under the influence of light only where water and oxygen are present, either in the fibre or in the atmosphere surrounding the fibre [1].

The present paper gives the results of a series of experiments designed to elucidate further the interrelation between the various factors involved. Certain theoretical deductions regarding the mechanism of the fading process are drawn, and these are compared with the views of Giles and co-workers [2,3].

2. EXPERIMENTAL

The difference in fastness to daylight of selected basic dyes on different fibres in various atmospheres has been examined by a method originally devised by Schwen [4].

The dyeings were exposed in glass tubes filled with the following gases— (1) dry oxygen, (2) moist oxygen, (3) dry nitrogen, (4) moist nitrogen, (5) control exposed in an open tube to normal atmosphere but under similar conditions.

The glass tubes used were made from "Jena'er Geräteglas", 1 metre long and 6·5 cm. in internal diameter, sealed at one end, near which there was a constriction making the lower end of the tube a container for liquids or dry chemicals. At the upper end of the tube was a ground glass joint occupying the full diameter of the tube. This joint was fitted with a ground glass attachment for filling the tube with the requisite gas and subsequently sealing.

The tubes were prepared in the following manner—

(1) The tube for light exposures in dry oxygen contained anhydrous calcium chloride.

(2) The tube for exposures in moist oxygen contained water.

(3) The tube for exposures in dry nitrogen contained a mixture of dry pyrogallol and dry potassium hydroxide.

(4) The tube for exposures in moist nitrogen contained a solution of pyrogallol and potassium hydroxide in water.

(5) The tube for exposures in a normal atmosphere was made from a cylinder of the same glass, having the same dimensions, but with full bore open to the air at both ends.

The dyeings were mounted on glass plates covered with black paper, the dyed patterns being 1·1 cm. wide by 5·5 cm. long and stuck to the black paper with a suitable adhesive (free from solvent and formaldehyde). This particular arrangement has the advantage that a larger number of patterns can be exposed at the same time and under the same conditions. A technique similar to the above was used by Cunliffe [5] for studying the effects of humidity and temperature on light fastness and by Agster and Holzinger [6] for studying the effect of light exposure on fibres.

The assessments of the exposed patterns were made against a blue scale (E.C.E.) simultaneously exposed in the open tube under normal atmospheric conditions. Light fastness figures were determined under the conditions of the Deutsche Echtheitskommission corresponding to E.C.E. by experienced personnel.

The commercial dyes were used in the form normally supplied, but the diazacyanine dyes were used as 100% laboratory products. All dyeings were prepared by their normal dyeing methods to a depth corresponding as exactly as possible to $\frac{1}{3}$ of the standard depth.

The exposures were made in the daylight fading station of BASF in Ludwigshafen. Test Series A was exposed from September 1955 to April 1956. Test Series B was exposed from August 1956 to September 1956.

Series B tests were conducted after Series A with similar but not identical dyes, in order to verify the trend of the results obtained in Test Series A. Each series of dyes contained members of the following four chemical classes— oxazine, methin, triphenylmethane, and diazacyanine. The dyes were applied to the following materials— cotton mordanted with Katanol O, viscose rayon, secondary cellulose acetate, silk, wool, and Orlon 42 (DuP) polyacrylonitrile fibre.

Test Series A was exposed to a full fade of 7 on the Blue Scale (E.C.E.) and Series B to a first fade of 6, the Blue Scale in question being that in the open tube.

Light fastness was assessed to quarter grades and also below a grading of 1. Because of the conditions of exposure the light fastness figures obtained in the open tube do not necessarily correspond to those given in the pattern cards.

3. ANALYSIS OF RESULTS

The values obtained (Tables I and II) show the following two broad effects—

(1) The light fastness of the dyeings of some basic dyes on various fibres depends to a large extent on the amount of oxygen present in the surrounding atmosphere.

The surprising fact is that exposure at a given humidity in nearly pure oxygen does not necessarily give the lower light fastness figure one would tend to expect. In several cases in both series of experiments the results obtained in the tubes containing nitrogen are worse than those obtained in oxygen.

This happens with the diazacyanine blue when dyed on cotton, cellulose acetate, and silk. When dyed on viscose rayon and wool the same result is obtained, but in these cases we observed that the colour of these dyeings had also changed (in hue only, not depth) in the covered, unexposed portions of the dyeing. With this dye we also found that under all the experimental conditions it was unaffected on Orlon at the end of the period of exposure and had a light fastness greater than 7. With the diazacyanine yellow the same tendency to lower light fastness when exposed in nitrogen was found on all fibres.

This tendency is observed also with one of our oxazine dyes— C.I. Basic Blue 3— but only on silk and wool. With the other oxazine dye— C.I. Basic Blue 12— this tendency is not observed, the worst result being given by exposure in oxygen.

(2) As anticipated, the light fastness of the dyeings depends upon the quantity of moisture in the tubes [11].

In general, exposure in moist oxygen, or moist nitrogen, gives worse results than exposure in the dry gases. With the strongly hydrophobic acrylic fibres, the influence of humidity in the nitrogen atmosphere tends to be small or insignificant. It may be noted that in one case light fastness in moist nitrogen is slightly better than in dry nitrogen, e.g. with Astra Violet FN on cellulose acetate.

4. DISCUSSION OF RESULTS

It is realised that the range of these experiments is rather restricted, particularly from the point of view of the number of dyes studied. Nevertheless, two dyes, viz. the two diazacyanine dyes, quite different in colour but structurally similar, exhibit properties different from those of most of the other dyes tested. The same "abnormal" properties are exhibited also by one of the two oxazine dyes under test, but only on wool and silk (i.e. protein fibres).

This fact, that two dyes with identical main structural groups fade more on all fibres in low partial pressures of oxygen, indicates that in this case the chemical constitution of the dye probably exerts the major influence on the process of fading.

Giles [2] has recently postulated that—

(1) In the main, differences in the physical form of the dye give differences in light fastness.

(2) The character of the substrate has an influence of similar magnitude. Fading on proteins

TABLE I

Test Series A

Dye	Material	Open Tube	Oxygen Moist	Oxygen Dry	Nitrogen Moist	Nitrogen Dry
C.I. Basic Blue 3 (oxazine)	Cotton	2–3	1	1–2	3–*4*	4–5
	Viscose rayon	2–*3*†	1–2	2	2	3
	Cellulose acetate	3–4	3	4–*5*	*3*–4	3–*4*
	Silk	2	2	3	1	2–*3*
	Wool	*1*–2	<1	2	<1	1–2
	Orlon	5–6	4	*4*–5	>7	>7
C.I. Basic Orange 22 (methin)	Cotton	1	⩽1	1	3–4	5
	Viscose rayon	*3*–4	2	3	4	5–6
	Cellulose acetate	5	4	4–5	5	6
	Silk	4–5	2–3	3	4–*5*	*5*–6
	Wool	4	2–3	3	4	4–5
	Orlon	5	3–4	*4*–5	6	6–7
C.I. Basic Blue 18 (triphenylmethane)	Cotton	1	⩽1	1	2–*3*	3
	Viscose rayon	2	⩽1	1–2	1	2
	Cellulose acetate	1–*2*	⩽1	2–3	5–6	6
	Silk	2–*3*	<1	2–3	4	5–6
	Wool	1–*2*	1	2–3	3	3–4
	Orlon	4	*1*–2	3–4	7	7
Diazacyanine blue	Cotton	1	<1	1–*2*	1	1
	Viscose rayon	1–2	2	*5*–6	•	•
	Cellulose acetate	4–5	6	7	3	5
	Silk	<1	1–2	3	⩽1	2
	Wool	•	2	2–3	•	•
	Orlon	>7	>7	>7	>7	>7

TABLE II

Test Series B

Dye	Material	Open Tube	Oxygen Moist	Oxygen Dry	Nitrogen Moist	Nitrogen Dry
C.I. Basic Blue 12 (oxazine)	Cotton	2	1	2–3	4–5	6
	Viscose rayon	1	⩽1	1–2	3–4	5
	Cellulose acetate	2–3	*1*–2	2–*3*	3–4	4–5
	Silk	1	(<)1‡	*1*–2	2–3	4–5
	Wool	2	1	2–3	*3*–4	4
	Orlon	3	2	2	>6	>6
Astra Violet FN Extra (C.I. 48075) (methin)	Cotton	<1	<1	*1*–2	*4*–5	*5*–6
	Viscose rayon	⩽1	⩽1	<1	*4*–5	5
	Cellulose acetate	⩽1	⩽1	<1	*5*–6	4–5
	Silk	⩽1	⩽1	<1	6	6
	Wool	⩽1	⩽1	<1	6	6
	Orlon	3	1	2	6	6
C.I. Basic Blue 26 (triphenylmethane)	Cotton	<1	<1	<1	1–*2*	*3*–4
	Viscose rayon	⩽1	⩽1	(<)<1	⩽1	<1
	Cellulose acetate	<1	<1	1	3–4	4
	Silk	<1	(<)<1	(<)1	2	3–*4*
	Wool	1	(<)<1	1	*2*–3	3–4
	Orlon	2–3	2	*2*–3	6	6
Diazacyanine yellow	Cotton	2–*3*	2–*3*	3	<1	2–*3*
	Viscose rayon	1–2	1–2	1–*2*	<1	1–2
	Cellulose acetate	6	5–6	6	4–*5*	6
	Silk	4	4	4–5	3–*4*	5
	Wool	4	4	4–*5*	3–*4*	4–5
	Orlon	>6	>6	>6	>6	>6

• Hue has changed in the covered portion of the dyeing. The fading on the uncovered portion may be graded as <1.

† Figures in italics indicate quarter grades: thus 3–4 means 3¼, whereas 3–*4* means 3¾.

‡ Parentheses indicate intermediate values: thus (<)<1 lies between <1 and ⩽1.

seems to be a reduction; on the other fibres it appears to be an oxidation [3, 7].

(3) The chemical constitution of the dye itself has, relative to these factors, much less significance.

In our experiments we have not examined the state of aggregation of the dyes.

Regarding the second of these postulates, we also find an appreciable influence of the fibre, but in our case not so clearly restricted to the contrast between protein and non-protein fibres [7].

Regarding the relative influence of chemical constitution on light fastness, we have in the instances cited above found this of some significance in the fading of the dye.

This contradiction to the arguments put forward by Giles following his statistical investigations into this matter seems more apparent than real. We would point out that all the light-fastness values in Giles's investigations are obtained under normal atmospheric conditions. In our investigations, however, we have "analysed" the light fastness under normal atmospheric conditions and expressed it in terms of several individual values. The normal light-fastness value is, as we have shown, obviously a composite value, and the relationship between the individual values seems to be governed by laws which are as yet unknown to us. The possibility that this composite value may follow the rules postulated by Giles is in no way contradicted by our results.

5. THEORY

In the following we give a cautious interpretation of the results obtained.

As a working hypothesis we assume that, with those dyeings which exhibit lower light fastness under the influence of a higher partial pressure of oxygen, at least a part of the reaction is due to photo-oxidation. Conversely, dyeings which exhibit lower light fastness under extremely low partial pressures of oxygen (i.e. in nitrogen) may be subjected, at least partly, to a reducing action, although it is very unlikely that the molecular nitrogen contained in our tubes could itself exert any reducing effect.

The most marked result is that, when applied to nearly all fibres used in our work, our two diazacyanines appear to be reduced; i.e. they fade more in the presence of nitrogen than in the presence of oxygen.

Furthermore, when exposed to light in nitrogen, the worst results were obtained in the presence of moisture, i.e. water. As it is unlikely that the nitrogen itself takes any part in the reaction, the question arises whether, perhaps, water can have some reducing action.

As is known, a fairly high amount of energy is necessary for the decomposition of water into its components, and only very short ultraviolet radiation can have this effect. Radiation of such short wavelength (179 mμ.) will not be present at the surface of our dyeings, as it is absorbed by the oxygen and the moisture of the air when sunlight passes through the atmosphere.

It may be relevant to consider the process of photosynthesis in plants, which, too, requires that water should be decomposed into its components [8]—

> According to van Niel's first four quanta theory, the following reaction may be postulated—
>
> $$4\ H_2O \xrightarrow{4\ h\nu} 4\ \{H\} + 4\ \{OH\} \qquad (1)$$
>
> The brackets indicate that the components and products of this reaction do not occur in the free state. Water is assumed to be attached to a molecular complex, which probably includes a sensitiser (chlorophyll), while the "primary reduction product" {H} and the "primary oxidation product" {OH} are taken up by unknown "acceptors". With free molecules, atoms, and radicals, reaction (1) would require less than 110 kcal./mole, i.e. more than twice the energy available in one quantum of red light. In order to make the primary reaction at all possible, the energy of association of the products, {H} and {OH}, with their acceptors, must be larger than that of water, by at least 70 kcal./mole. Instead of representing the elementary photochemical process as a decomposition of water, as it appears in equation (1), it may be useful to emphasise its character as an "oxidation–reduction" with water (or a water–acceptor complex) in the part of the reductant and an intermediary hydrogen acceptor in the part of the oxidant. In this case we may write using the symbols introduced in equation (1)—
>
> $$4\ \{H_2O\} + 4\ Z \xrightarrow{4\ h\nu} 4\ \{OH\} + 4\ HZ \qquad (2)$$
>
> We have further to assume the dismutation of the oxidation product {OH} into water and oxygen. The latter can occur either directly, or through the intermediary of biradicals (peroxides $\{OH\}_2$ . . .).

The reactions mentioned show that in daylight and in the presence of certain acceptors water can have both a reducing and at the same time an oxidising effect.

The results of our work can thus be interpreted correctly only if we assume the presence of water under all our conditions. Fortunately, under our experimental conditions, although the partial pressures of oxygen and water were very low, they were not zero. One reason for this was that the dyeings were conditioned in the air and not thoroughly dried. Although we have referred to "dry oxygen" and "dry nitrogen", we must reckon with the presence in our dyeings of the small amounts of water that remain in fibres under normal drying conditions.

It follows that one of the conditions necessary for the above equations to hold, viz. the presence of water, was satisfied in all our exposures. The other condition, viz. the presence of acceptors, was probably satisfied also with the diazacyanine dyes. This is confirmed by the fact that both dyes exhibit behaviour similar in one respect to that of chlorophyll, in that they fade in the absence of oxygen, whilst in the presence of oxygen no fading occurs. We are inclined to believe that this behaviour is associated with the structure of the dyes. The general formula of diazacyanines is—

$$\rangle\overset{+}{N}{:}CH{\cdot}N{:}N{\cdot}[C{:}C]_n{\cdot}N\langle$$

It seems, therefore, that in the exposure of our diazacyanines a reaction according to equation (2) has taken place. This means that, when exposed to daylight and in the presence of a limited amount of oxygen only, both diazacyanine dyes accept the hydrogen of the water. Consequently, they are reduced, whilst the water itself is oxidised to {OH}. This oxidation product {OH} will react subsequently as described above, depending

on whether or not an acceptor for the OH is present or whether the reaction leads to formation of H_2O_2 or H_2O and O_2.

In a personal communication received from Dr. C. H. Giles, he suggests that this section could also be discussed by reference to the paper of Hillson and Rideal [9]—

> These authors found that, when the dye is illuminated in the presence of water, the excited form combines with water to form a free radical and hydrogen atom as follows—
>
> $$D \xrightarrow{h\nu} D^* \quad D^* + HOH \longrightarrow DOH + H\text{-}$$
>
> Normally, therefore, moisture is required and also oxygen to combine with hydrogen and so sustain the reaction. It seems to me that if, as in the case of the two cyanine dyes used here, the dye itself is much more readily reduced than oxidised, then the hydrogen may attack another molecule of dye instead of O_2.

The main difference between the two theories is the question of the photochemical primary reaction. We understand that Rabinowitch and van Niel regard the dye as "sensitiser" or "acceptor", whilst Hillson and Rideal are of the opinion that the dye molecules form "free radicals" under the influence of light and in the presence of water. Essentially, it is a question of the energy changes involved, and the question does not lie within the scope of this paper. Both of these theories, however, lead eventually to the same results as far as the evaluation of our results is concerned.

6. CONCLUSIONS

We may now state that, under all our conditions, our diazacyanine dyes are primarily reduced in the fading process and the other dyes in our tests are in general probably oxidised. This fact is of special interest, for in solution both diazacyanines are quite sensitive to reduction. The other dyes tested are sensitive rather to oxidation in solution, especially the two triphenylmethane dyes.

It is most likely that the extent of the reduction of the two diazacyanine dyes depends on the quantity of water present in the fibre. In this connection it was interesting to observe that dyeings of these dyes on the hydrophobic Orlon are affected only slightly or not at all by the reduction process.

It is probable that reduction of the diazacyanines on hydrophobic fibres occurs only during daylight illumination, and even then only on the surface of the dye particle, where it is in direct contact with moisture. Active hydrogen can occur only at those places where it is taken up by an acceptor, or in other words, where a reaction takes place immediately.

With the other dyes examined, the oxidation effect, i.e. the fading effect, on the hydrophobic Orlon is much more pronounced. This oxidation process may possibly be caused by quite different reactions from those discussed above. For example, it is well known that during the illumination of many undyed fibres the production of hydrogen peroxide is observed [10]. It may thus be assumed that the oxidation observed with the triphenylmethane, oxazine, and methin dyes is caused at least partly by hydrogen peroxide.

This peroxide dissolves, of course, in water, and oxidation of our dyes can, therefore, occur whenever and wherever moisture is present. In other words, oxidation of the dye itself is not dependent on the direct and simultaneous influence of light: it can continue in the dark. The moisture contained in the fibre can be regarded as a "reservoir" for the hydrogen peroxide formed during exposure, but we cannot conceive of any such "reservoir" for the reducing agent, active hydrogen. These ideas appear to be confirmed by the fact that dyes which are generally susceptible to reduction are practically unaffected by reduction when applied to Orlon; whereas those dyes which are susceptible to oxidation are influenced very strongly by oxidation, even when applied to the hydrophobic Orlon.

Finally, it appears that the first step towards oxidation or reduction causing the fading of a dyeing possibly depends on the ability of the dye to act as an acceptor.

Quite a number of questions remain unanswered, and we realise that in our endeavours to interpret the results of our experimental work we have made assumptions for which we have no real proof.

* * *

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(*MS. received 3rd May 1958*)

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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Dyeing of Wool at Room Temperature

The main disadvantages of conventional processes for dyeing wool lie in the lengthy periods for which the wool is subjected to high temperatures. These factors contribute a large part to the cost of dyeing and are a potential source of damage to the wool. Recent investigations by Stevens and Peters [1] and by Kärrholm and Lindberg [2] have shown that both the time and the temperature necessary for dyeing wool may be considerably reduced by the addition of a small quantity of a sparingly miscible solvent to the dyebath. The temperatures required for this system of dyeing are in the vicinity of 50–60°C., and satisfactory dyeing is attained in 15–30 min.

Some experiments by the writer [3] now demonstrate that it is possible to dye wool at room temperature in a matter of 1–2 min. This may be achieved by using a dyebath in which the dye is dissolved in formic acid instead of water. The more nearly anhydrous the formic acid, the speedier is the dye uptake, but commercial 85–90% formic acid is satisfactory. It is clear that such a process could greatly simplify the problems involved in the development of continuous dyeing techniques, and further studies are being pursued to that end.

The effectiveness of the process has been tested on a number of commercial dye samples ready to hand in this laboratory, and from these tests it appears that the procedure is applicable to dyes of the acid and milling acid types and also to the neutral-dyeing 2 : 1 metal-complex dyes. Preliminary experiments also show that the Cibacron (Ciba) dyes are taken up by wool from formic acid solutions. As might be expected, the process is generally inapplicable to dyes of a basic character. Some dyes take on a different hue in formic acid solution, probably as a result of an "indicator" tautomerism, but the dyed wool reverts to the normal colour associated with the dye on washing with water.

As yet, no other solvent has been found which even approaches the effectiveness of formic acid. It seems likely that a favourable combination of several factors is responsible for its apparently unique properties. Firstly, the wool fibre is considerably more swollen in formic acid than in water [4], thus lowering the barrier to diffusion of dye through the fibre. Secondly, the dielectric constant of formic acid is relatively high (58·5 [5]), so that ionisation of the wool protein and dye molecules can take place. Thirdly, it is probable that in formic acid solutions the sulpho groups in the dye molecule remain ionised, while the carboxyl groups on the wool keratin are un-ionised, so that the protein shows its maximum acid-binding capacity, and hence strong Coulomb attractive forces can operate between the dye and the wool protein. It is also suspected that the dye molecules are less aggregated in formic acid than in water, thus increasing the diffusion rate of dye into the fibre. This idea is being tested by further experiments.

An advantage of the process is that the wool is wetted out by the formic acid immediately upon entering the dyebath without the addition of wetting agents which is usual in conventional dyeing procedures. Microscopic examination of cross-sections of wool dyed from formic acid baths has shown that penetration is uniform throughout the fibre (G. E. Rogers, *personal communication*). Exhaustion of the dyebath is attained in this process only for pale colours. For greater depths a considerable excess of dye must often remain in the dyebath. However, for the continuous dyeing process envisaged this is not a disadvantage. It has been noticed that the addition of inorganic salts to the solution of dye in formic acid inhibits uptake of dye by the wool to a very marked degree, although the quantities of salt normally present in the commercial dyes used have so far presented no difficulty.

The main application of this process appears to be for the dyeing of loose wool and tops. For fabric composed of high-twist yarns penetration of the dye is poor. This is due to the swelling of the surface fibres preventing ingress of the dye liquor to the inner portions of the yarn. On the other hand, pieces may be printed with formic-acid solutions of dye thickened with Cellofas B (ICI) or other agents. This process eliminates the necessity for either chlorinating the wool or steaming the printed fabric.

The commercial success of a process of this type must depend on the cost of the formic acid and the ease of its recovery. Work in progress in this laboratory suggests that the recovery of the formic acid may be achieved relatively easily. Against these costs must be set the advantages of a very rapid process which may be adapted for continuous dyeing as well as the savings in steam and additives to the dyebath which are required in the normal process. Furthermore, preliminary tests suggest that damage to the wool is less than that caused by conventional aqueous dyeing processes.

The results of more detailed experiments will be reported at a later date.

The writer's thanks are due to Dr. F. G. Lennox and Mr. T. A. Pressley of this laboratory for much technical advice and stimulus.

Note added in Proof

It has been pointed out to the writer that Hall [6] has already made some observations on the effect of formic acid and acetic acid on the uptake of dyes by wool at room temperature. For example, exhaustion of Kiton Red S (2% on weight of wool) has been found to be almost complete at room temperature from dyebaths containing more than about 5% (wt./vol.) formic acid. It is also stated that "similar results were obtained with acetic acid in place of formic acid, the concentrations

required for equal degrees of exhaustion being slightly higher in the former case". Unfortunately, Hall does not state his time of dyeing, but it is clear from a few experiments by the writer that the uptake values given were probably obtained at equilibrium, requiring long periods of time unless the concentration of formic acid is very high. Since a high rate of uptake is essential for the continuous process suggested in the present letter, the long-term values of Hall have little bearing on this aspect of the work. As an example, it has been found that the uptake of Kiton Red S from a 90% (wt./vol.) formic acid solution is greater in 1 min. than that from a 25% (wt./vol.) formic acid or a 50% (wt./vol.) acetic acid solution after 2 hr.

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30th October 1958

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Notes

Meetings of Council and Committees

January

Council— 7th
Finance and General Purposes— 22nd
Publications— 20th
Fastness Tests Co-ordinating— 29th
Terms and Definitions— 16th
Colour Index Editorial Board— 20th
Data on Identification of Dyes on Textile Fibres— 2nd
International Relations— 29th
Historical Records— 30th.

Death

We regret to report the loss by death of Mr. F. Arend.

Bradford Junior Branch Diamond Jubilee

The Diamond Jubilee of the foundation in 1898 of the Bradford Junior Branch of the Society was celebrated with a Dinner and Dance held at the Victoria Hotel, Bradford, on Friday, 5th December 1958, attended by over a hundred members and guests. The toast to the Branch was proposed by Dr. C. M. Whittaker, a Past President of the Society, who stressed the contribution of individual effort rather than teamwork and organisation in producing major advances in science and technology. The Chairman of the Branch, Mr. R. S. Harding, when responding to the toast read a telegram from the President of the Society, Mr. John Boulton, in which he expressed his regret at being unable to be present and congratulated the "Society's eldest grandchild" on its achievement. The toast to the guests was proposed by Mr. E. N. Wood, a member of the Bradford Junior Branch Committee, and responded to by Mr. Fred Smith, a Past President of the Society and a former chairman of the Bradford Junior Branch.

Below is a reproduction of Professor Walter M. Gardner's notice, dated 10th February 1898, convening a meeting of students at the Bradford Technical College to form the Society's first Junior Branch.

Society of Dyers & Colourists.

It has been suggested that the junior members of the Society should arrange to hold a few meetings each session at which discussions may take place, or addresses may be given, in a less formal manner than at the ordinary meetings of the Society.

With a view to a discussion of this project a meeting will be held in the Dyehouse Lecture room next Thursday (Feb. 17th) at 8·30 p.m., to which all students and their friends, whether members of the Society or not, are cordially invited.

Feby. 10th 1898.

Walter M. Gardner.

A.S.D.C. Examination 1959

The annual examination for the Associateship of the Society of Dyers and Colourists will be held on Thursday, Friday, and Saturday, 4th, 5th, and 6th June 1959. Candidates should note that Papers A and B may be taken together, followed by C, D, and either E(i) or E(ii); or Papers A, B, and C may be taken together, followed by D and either E(i) or E(ii). Candidates should indicate the special branch of tinctorial technology in which they desire to be examined and state whether they intend to take the examination in one or two stages and which of the two Papers E(i) or E(ii) they will take.

Application forms are obtainable from the offices of the Society and must be completed and returned not later than Saturday, 28th February 1959.

Society of Chemical Industry Paper and Textile Chemicals Group

The inaugural meeting of the Group was held in Manchester on 30th October 1958. Dr. A. K. Mills (Vice-president of the S.C.I.) opened the meeting, and Dr. H. A. Thomas (Chairman of the Group) presided.

Changes in Permitted Food Dyes

The Government is prepared to consider proposals for individual dyes to be added to or removed from the permitted list now embodied in the Colouring Matter in Food Regulations, 1957 (S.I. No. 1066) and the Colouring Matter in Food (Scotland) Regulations, 1957 (S.I. No. 1123). The Government intends that the list as a whole should be reviewed in not more than five years' time, but the following procedure will enable the list to be modified in the interim should new developments in dye manufacture or food processing, or fresh evidence relating to the safety or otherwise of dyes, make this desirable.

All such proposals should be addressed in the first instance to the Assistant Secretary, Food Standards, Hygiene and Slaughterhouse Policy Division, Ministry of Agriculture, Fisheries, and Food, London S.W.1, or to the Secretary, Department of Health for Scotland, St. Andrew's House, Edinburgh 1.

Proposals for dyes to be added to the list of permitted dyes should include information on the following points—

(*a*) Identification of the dye (the *Colour Index* number would be sufficient)

(*b*) Properties and proposed uses

(*c*) Evidence of freedom from health hazard.

Notes on these points and an indication of the form in which the information should be provided are given in *Chem. and Ind.*, (45), 1472 (8 Nov. 1958).

Proposals for the deletion of dyes should state the reasons. Where these have to do with the safety of the dye, details of the evidence should be given.

Food Dyes in Holland

A decree limiting the types of colorants which may be used in the preparation of foodstuffs and beverages will come into force in Holland in August 1959. The text of the decree may be seen on application to the Export Services Branch (Tariff Section), Board of Trade, Room 722, Lacon House, Theobalds Road, London W.C.1.

Food Dyes in West Germany

A new law has been passed by the Bundestag which prohibits the addition to foods of any substance "without nutritional value" unless it has been specifically declared safe. Twenty-five dyes have already been approved for use in food. Previously in Germany a substance could be added to food unless it had been declared to be dangerous.

Speckled Paint

According to *Du Pont Magazine*, **52**, 10 (Aug.–Sept. 1958) "polka dot paint" is now available in the U.S.A. The method of manufacture is based on *USP* 2,591,904 (John C. Zola): a water-insoluble cellulose nitrate lacquer is stirred into water containing a stabilising agent, yielding a uniform dispersion of droplets, whose size and shape can be varied by adjusting the temperature or speed of mixing. The dispersion can be either applied directly or blended with an unlimited number of other dispersions of different colours; also, glossy finishes can be mixed with flat ones. Multicolour lacquers are claimed to have very good hiding power.

Closure of the Last Factory in England to bleach Flax Yarn

On 6th December 1958 the 113-years old Hunslet Linen Works of Richard Buckton & Son Ltd., Goodman Street, Leeds, the last firm in England to bleach flax yarn, closed down. Throughout the firm's history the bleaching was done in the huge stone troughs installed in 1845 by the firm's founder, Mr. Richard Buckton. C. O. CLARK

Colour Index

Celebration Dinner

A Dinner to celebrate the publication of the Second Edition of the *Colour Index* was held on Monday evening, 15th December 1958, at the Victoria Hotel, Bradford. Mr. John Boulton presided, and about eighty representatives of dyemaking and dye-using firms and others who had contributed to the work were present. The menu card was suitably adorned with hexagons.

"THOSE WHO HAVE CONTRIBUTED TO THE WORK"

Mr. JOHN BOULTON (*President of the Society*), in proposing this toast, said: I am not going to talk about the Second Edition of the *Colour Index* as such. Even to summarise the detail that has gone into the four volumes of this great and complicated undertaking, much less to mention the editorial, administrative, and financial problems in which Council has been and is still involved, would exhaust you long before I had exhausted the topic.

Observing the four magnificent tomes, one is struck immediately with the great expansion in our available knowledge in the last two decades. Where one volume sufficed, four are now needed. It is one of the more telling characteristics of our civilisation that its book population should grow at an ever more sharply increasing rate, and no topic under the sun should be without its book. On the subject of dyes, their manufacture, and their use, this great work is the book, and will be so for a very long time to come.

In tonight's gathering, and in the purpose for which we are gathered, there is a synthesis of all that is best in our Society. The Society of Dyers and Colourists is, vis-à-vis similar organisations in associated fields, in an enviable position, and has built up a much envied reputation. That reputation is not based entirely upon the high technical and scientific standards of our members.

What marks the Society as happily unique is the outstanding excellence of its collective effort. All that we do—the *Journal*, the Symposia, the Summer Schools, Fastness Tests and similar sponsored work, not to mention the Diplomas and Examinations, are only some of the visible consequences of that smooth collective effort.

The Society makes its impact as a result of voluntary work, impossible to estimate, put in week by week, year by year, and, for many of you here, decade by decade, by busy people in their own homes, in the Society's meeting places, and in the laboratories and offices of their firms. All that is implied in this generous and selfless gift of time, one's own time and one's firm's time, provides the real wealth of our Society. The use to which that wealth can be put by a corporate body, conscious as we are of our richness, is most wonderfully exemplified in the *Colour Index*.

It is not remotely possible to calculate even the order of man-hours that have gone into the completion of the Second Edition, but they represent a tremendous gift to the Society and to the dyemaking and dye-using industries, whose techniques and advancement are our concern.

My task tonight is to offer for the Society, in such modest ways as lie within our power, thanks on behalf of all who will benefit from the appearance of the Second Edition. The production of the *Colour Index* was a campaign which called for tactics, strategy, and diplomacy for its successful conclusion. Mr. John Barritt and his Editorial Panel were the general staff and, as such, deserve our first congratulations and respect. Then, all those friends and colleagues who helped the Panel through the tedium of compilation and proofreading.

Speaking now as the President of the Society, I extend deep gratitude to those of you here who represent industrial companies, large and small, dye manufacturers and dye users. You have, heedless of the huge salaries you no doubt pay your scientific staff, of the red items on the balance sheet, and of the anguished cries of shareholders, encouraged your colleagues who have involved themselves in this work to pursue that involvement without stint, let, or hindrance. I must similarly thank the research associations represented here for the contributions of their members at many levels, and also the Oil and Colour Chemists' Association.

Here tonight, then, are leaders in the dyemaking and dye-using industries, scientists and technologists from industry, from research associations, and from universities, all of us members of the Society and all met to celebrate a common effort which has been, as I said at the beginning, a synthesis of all that is best in our Society and in the industries themselves.

Not even a Society so generously endowed as ours with other people's time, and the labour of its members, can work without a permanent staff. I must mention the heavy burden of extra work that has fallen on Dr. C. J. W. Hooper and Mr. J. W. Nicholls and offer to them and the staff your best thanks for their devoted work.

Finally to our printers. One of the distinguishing features of the Second Edition is the beauty of its production. The volumes are a pleasure to handle, and the printed pages a pleasure to behold. In thanking Messrs. Chorley & Pickersgill Ltd. and Percy Lund, Humphries & Co. Ltd. for their decisive contribution to the finished work, I would say, with Shakespeare, "That book, in many's eyes, doth share the glory".

I cannot sit down without some mention of the part played by, first of all, the American Association of Textile Chemists and Colorists, who will no doubt be celebrating a joint achievement in their own way, and those overseas companies from Switzerland, France, and Germany especially, who have made important contributions.

Mr. JOHN BARRITT (formerly *Chairman of the "Colour Index" Editorial Panel*), in response, said: The toast, "Those who have contributed to the work", is a very wide one, and I cannot attempt to cover it all, so you will perhaps forgive me if I tend to concentrate on the work of the *Colour Index* Editorial Panel.

I should like to say first how sorry we are that Mr. R. J. Hannay is unable to be with us tonight. He is making good progress, and sends his greetings.

The first meeting of the Panel was held on 5th October 1945 in Manchester with Mr. H. H. Bowen in the chair; the 168th and final meeting was held on 20th August 1958 at the Wool Industries Research Association in Leeds, when I was in the chair.

Thirteen years is a long time, and this reminds me of the remark made recently by the Archbishop of Canterbury when a Commission was to be appointed for the revision of the Psalter. He said: "See that your members are under forty or they will not see the end of the work". I will not be so tactless as to refer in detail to the ages of the Panel, but I think they would generally be somewhat above the figure set by the Archbishop. However, I think I can mention that one of our members, Dr. H. H. Hodgson, joined the Panel after he had received his post-war credits and was, when well over the three-score years and ten, our liveliest raconteur during our luncheons.

During our thirteen years we did lose Mr. A. Shepherdson in the very early days, but otherwise we have all come through, though some fell by the wayside. If I were to be cynical I could say that maybe they saw the light, but I can assure you that they all had compelling reasons for leaving the Panel. Those remaining have become greyer and their sight has become weaker.

Three of us—Mr. H. Blackshaw, Mr. A. Thomson, and myself—were original members and stayed the course. Actually, in point of attendance, Mr. Blackshaw and Mr. Thomson won the marathon, because they attended the first two meetings, which I could not attend for technical reasons connected with my being allowed to take part in this work.

I would like to pay a tribute here to Mr. Bowen, who retired from the Chairmanship in 1949. He started us on the right lines and carried out, in his own inimitable way, very many delicate negotiations in the early stages of forming the Panel.

At our final meeting at Torridon we all signed the report [see p. 112] for submission to Council which gives an account of our work and makes suggestions for the future. I will not bore you with all the details of the report, other than to say that this Second Edition contains about three times as much material as the First Edition, which was published in 1924, and contains 3,595 disclosed constitutions and no loss than 31,500 commercial names.

You, Mr. President, have thanked the organisations which permitted members of the Panel to take part in this work, but, knowing how much time we have had to devote to the task, I, too, would like to say on behalf of the Panel how we appreciate the readiness with which our employers have permitted us to do this work. Very rarely will such blank cheques be signed.

Now I wish to pay tribute to a number of non-members of the Panel who have given great assistance in compiling this work. Naturally, my first thought is of Dr. F. F. Elsworth for his valuable help in connection with the acid dyes. Then we have Dr. B. C. Bhattacharya of India, who helped with natural organic colouring matters, Dr. E. J. Cross, who helped in revision of the azo dye section, Messrs. W. E. Wood and T. Z. Wozniczka, who compiled the section on sulphur dyes, and the Oil and Colour Chemists' Association, who helped with the solvent dye and pigment sections.

The Panel is full of admiration for the work of both the printers—Messrs. Chorley & Pickersgill Ltd. and Percy Lund, Humphries & Co. Ltd.—and especially the skill of the former in dealing with the complicated formulae of Volume 3—there are few printing houses who could tackle such a job.

When we started this work we did not envisage that it would take so long, and I recall that somewhere about 1950 some member said how appropriate it would be if we could publish in 1956, the year of the Perkin Centenary. At that time our considered view was that we could not spin out the job until 1956. However, the material came in slowly, there were inherent delays in dealing with our American partners, and we all found it more and more difficult to take time from our normal work. We have all had to spend many hours doing home work, and I recall that one weekend I did quite a bit of work in preparing urgent proofs for the printer at the Majestic Hotel in Harrogate. I must confess that, on this occasion, Mr. Foster Beaver had invited me to attend a week-end conference for spinners, and I sneaked off as early as possible to do this work.

Now I would like to say a word on more general matters, and I draw on the early years of the project, when I was still Honorary Secretary of the Society. Everything has not been plain sailing throughout these years, and I remember that when Dr. C. M. Whittaker was President, we were in serious difficulties with the AATCC. The Americans were proposing to apply a veto on published information, and our attitude was that we should make use of any published information, even if it referred incidentally to American dyes. Arising from this we had the famous meeting in June 1948 at Buxton with the then President of the AATCC (Mr. J. R. Bonnar) and Mr. W. D. Appel, where we very readily reached agreement: in fact, the Buxton Agreement was made, which disposed of the veto. Perhaps the greatest difficulty which has been experienced is the lack of direct contact with our American partners. Whenever we have met them and been able to discuss our problems, difficulties have disappeared, and this emphasises that written communication is a poor substitute for personal contact. It is sad to recall that, of the five Society representatives at that meeting, only Mr. Blackshaw, Dr. Hooper, and myself remain; Messrs. F. L. Goodall and H. Jennison, who did

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HAROLD HENRY BOWEN F.S.D.C. (Bar)

Professor WILLIAM BRADLEY D.SC. PH.D. F.R.I.C. F.S.D.C.

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DIPLOMA OF THANKS

ALL who have contributed individually or collectively to the production of the *Colour Index*

John Barritt

Harold Blackshaw

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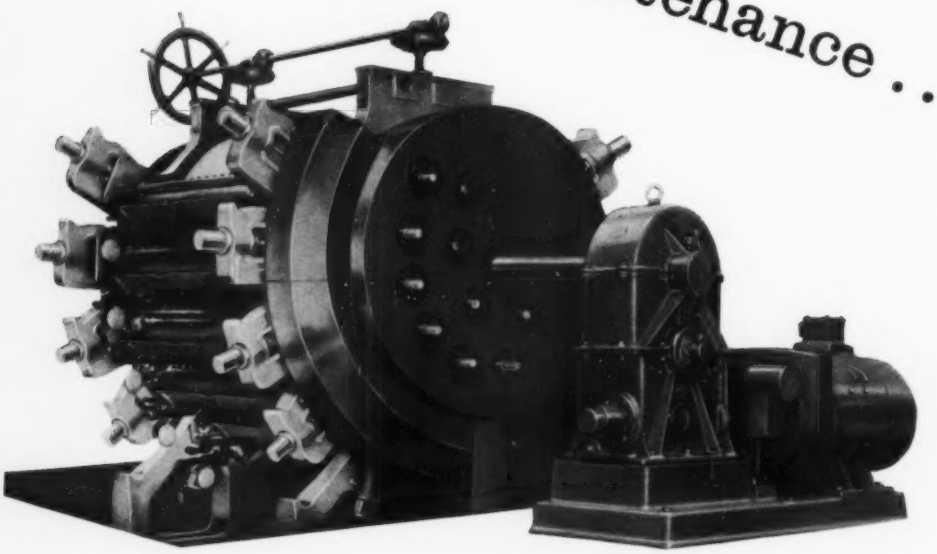

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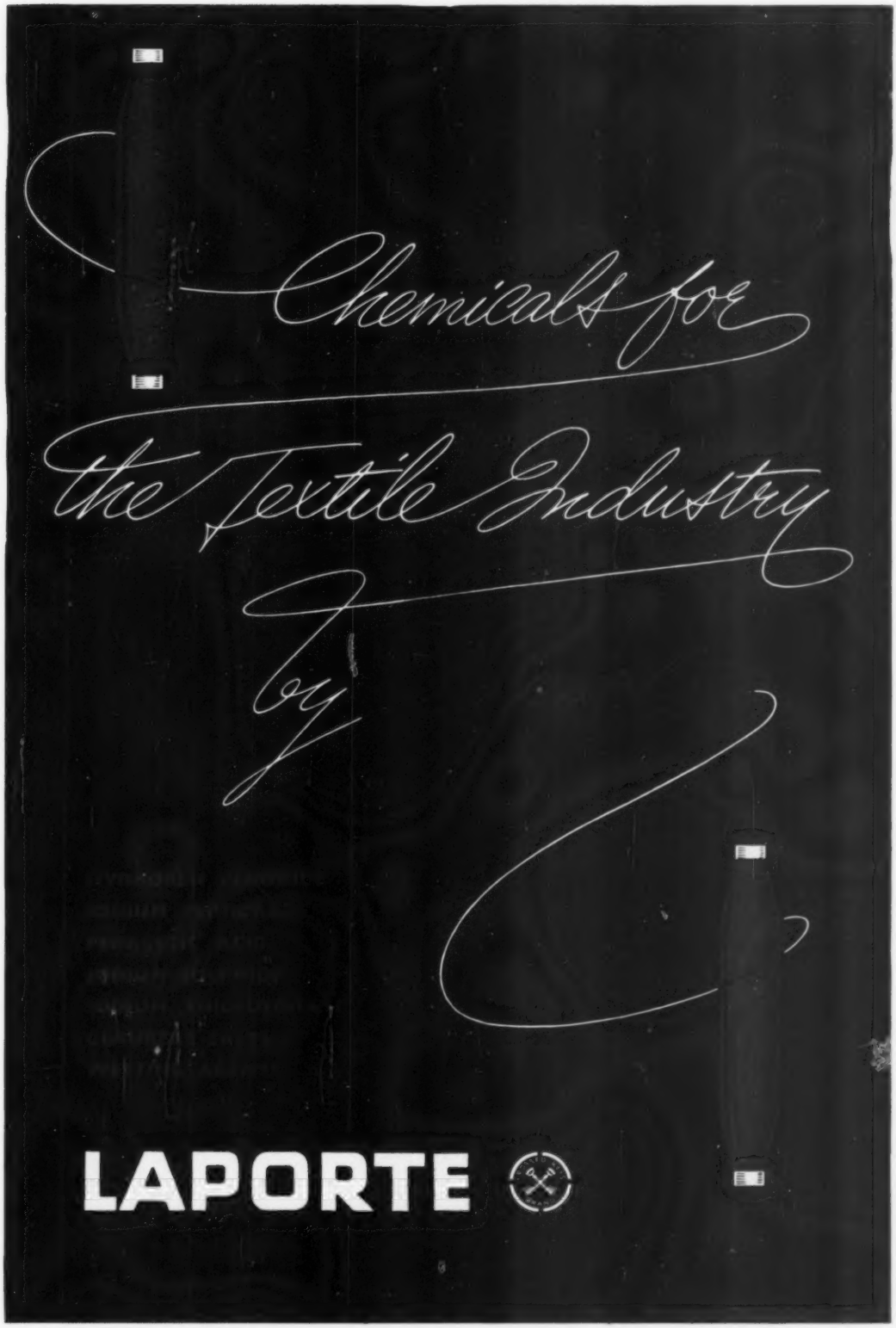

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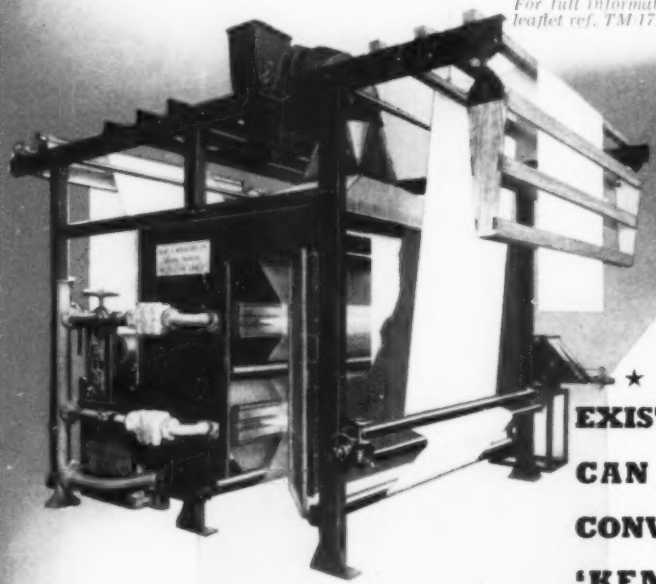

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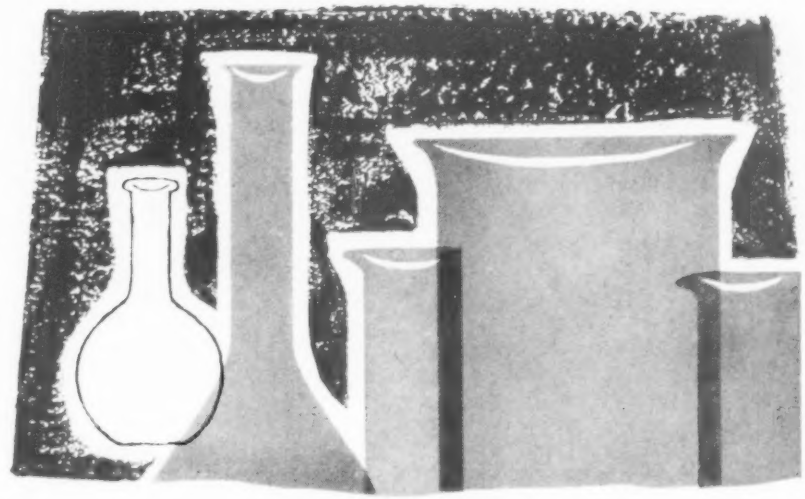

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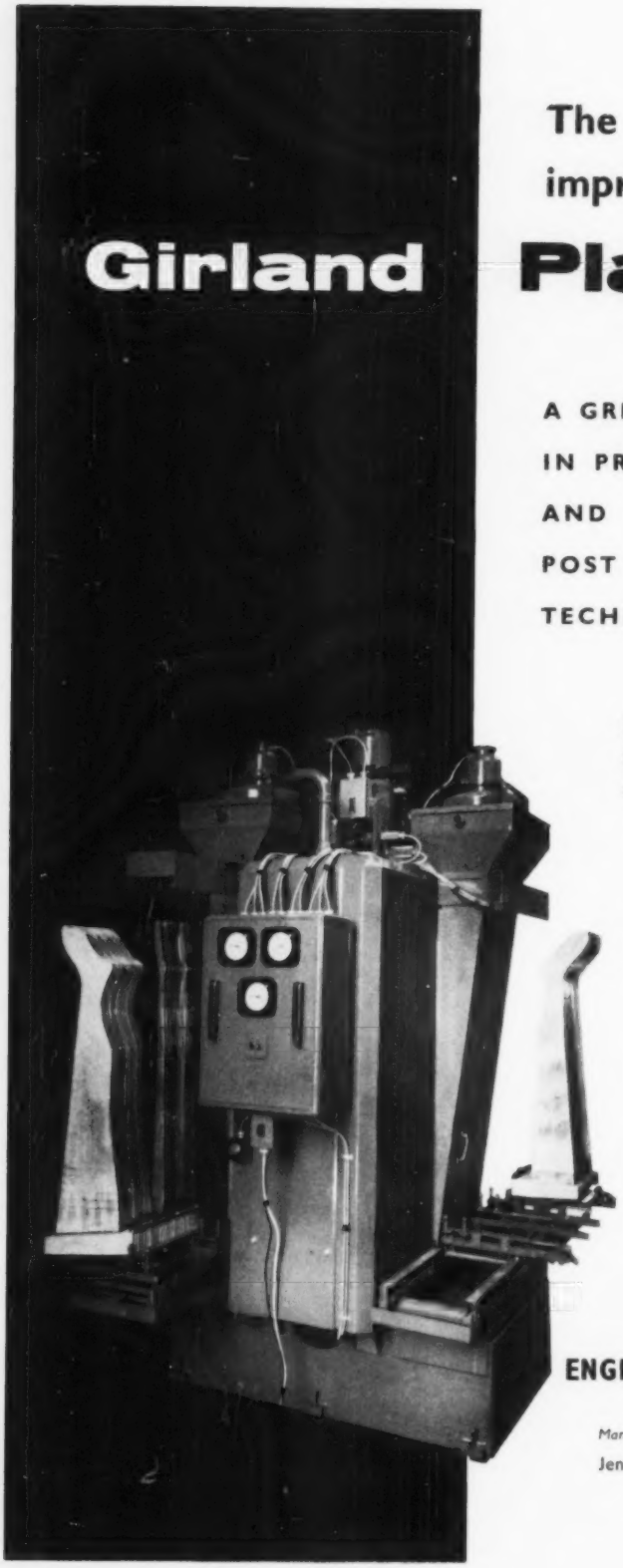

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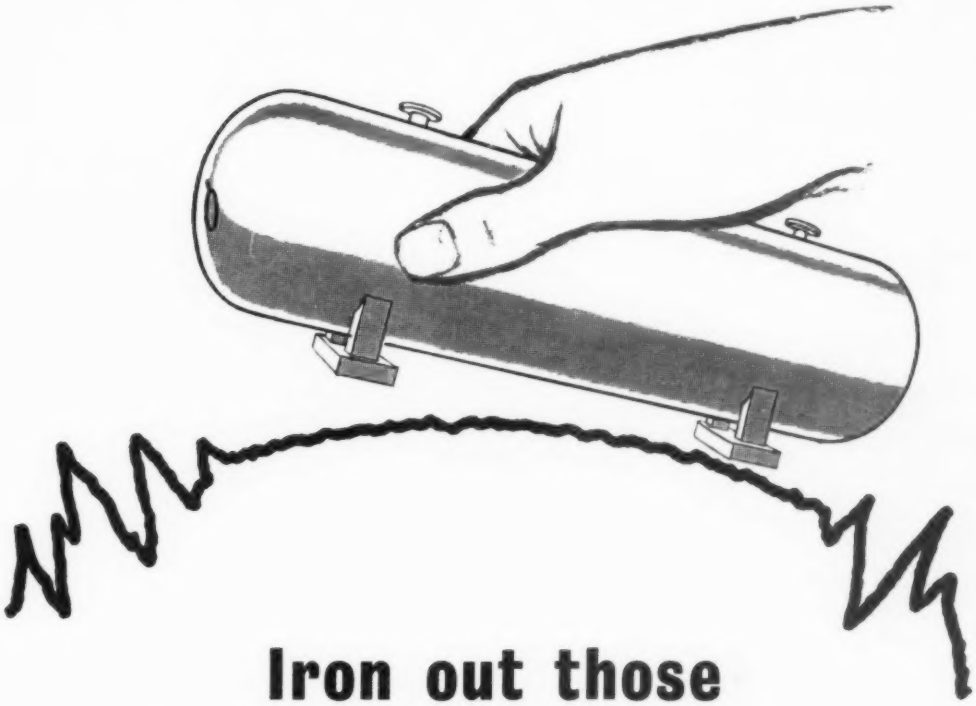

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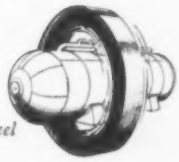

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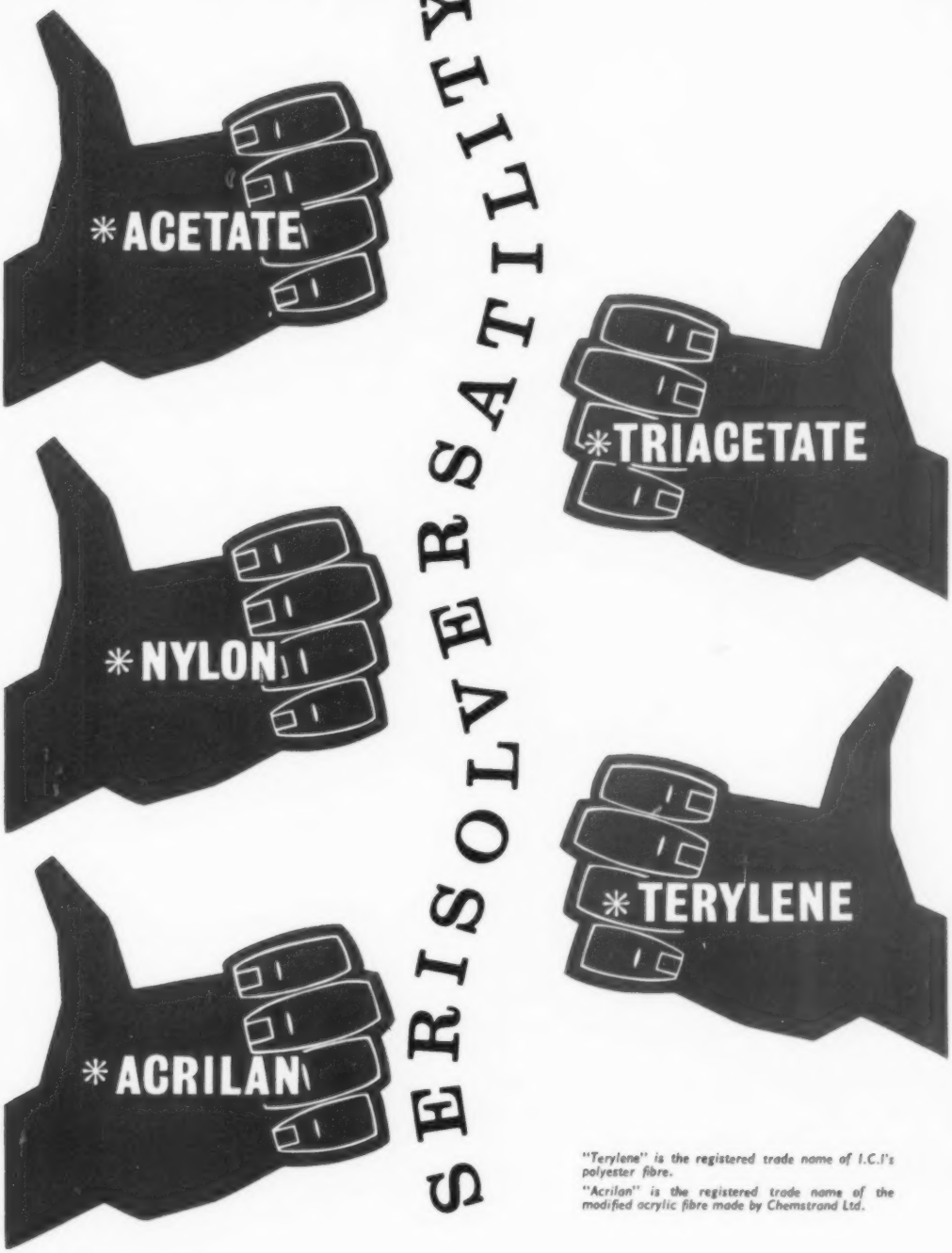

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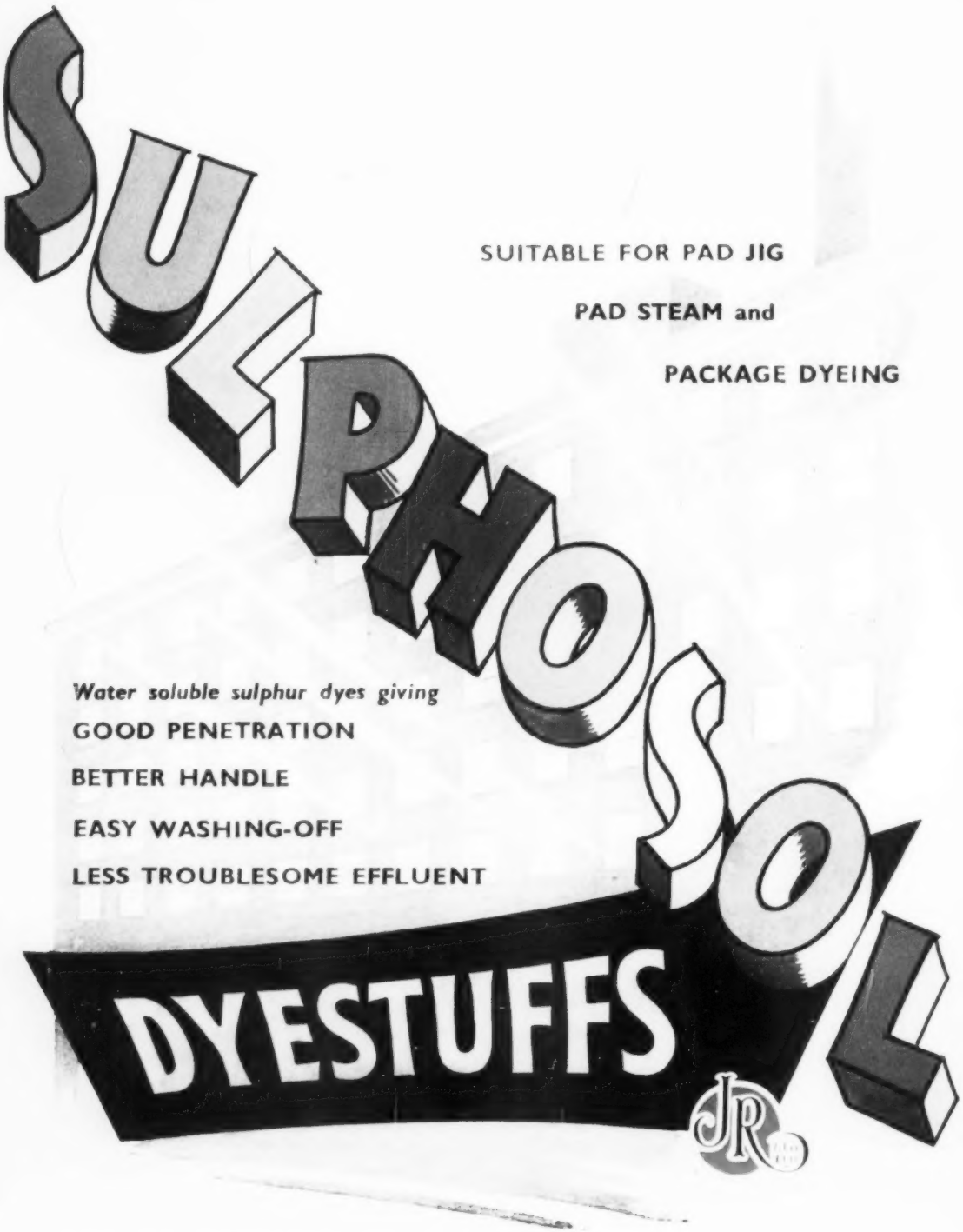

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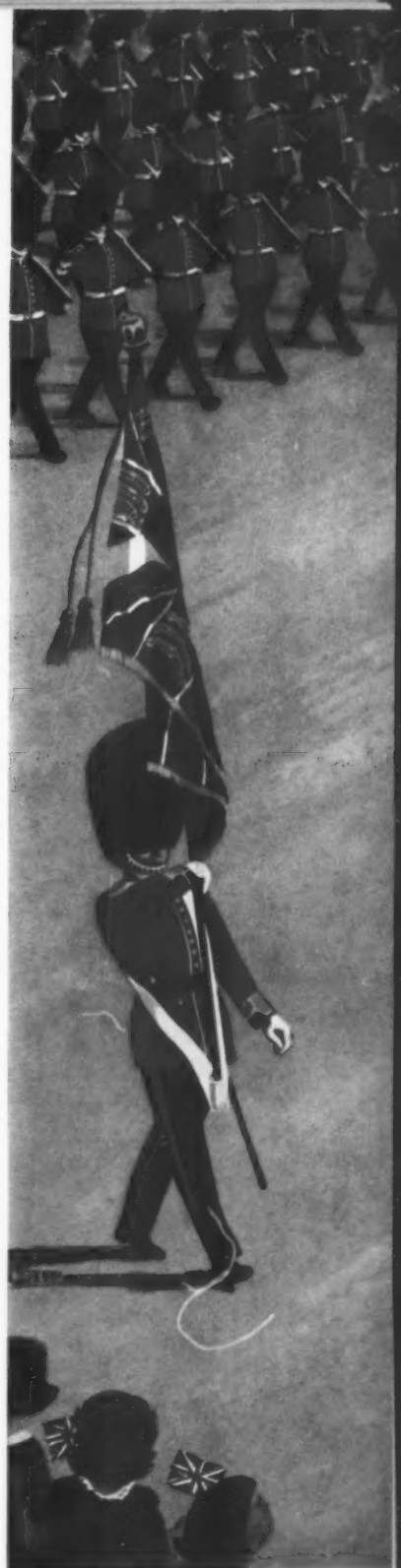

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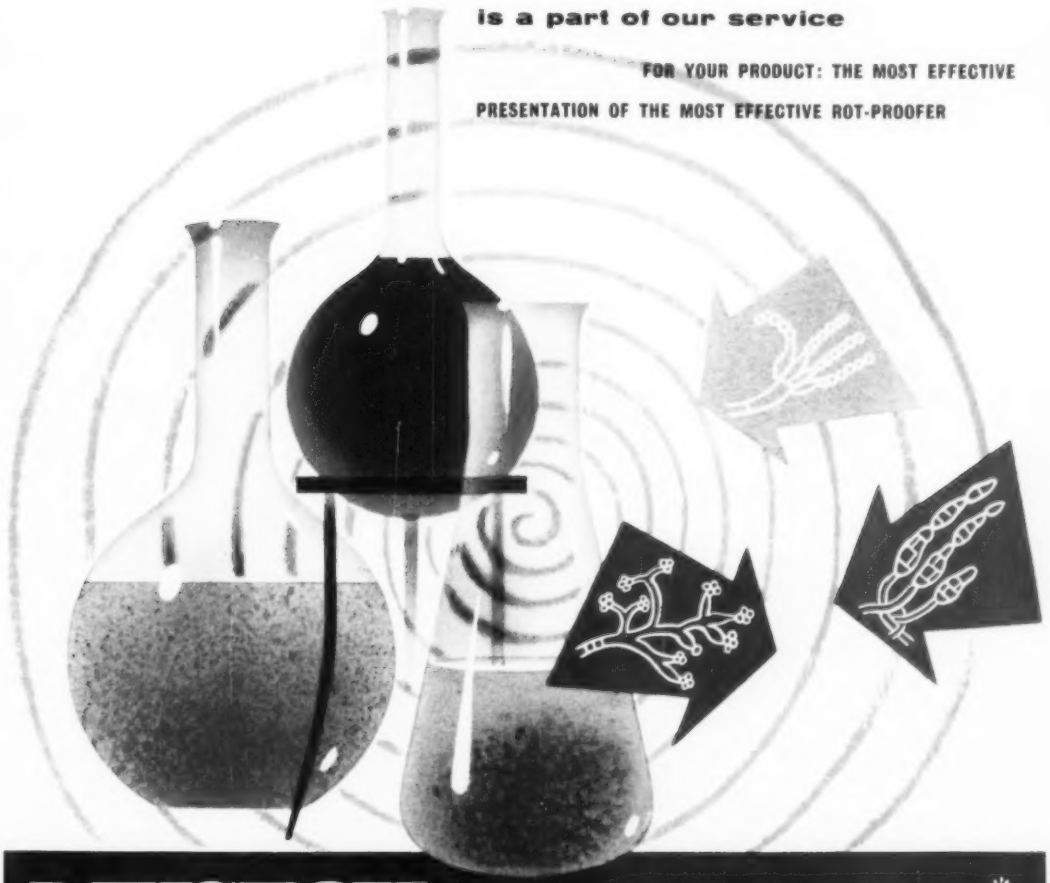

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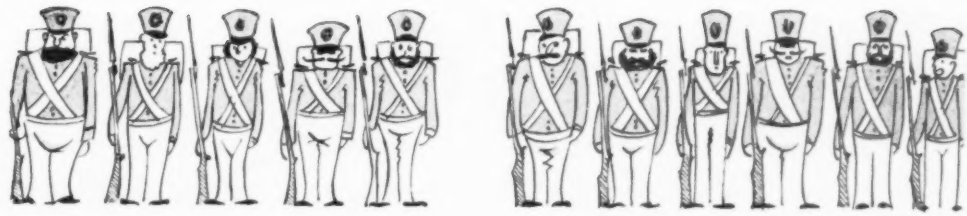

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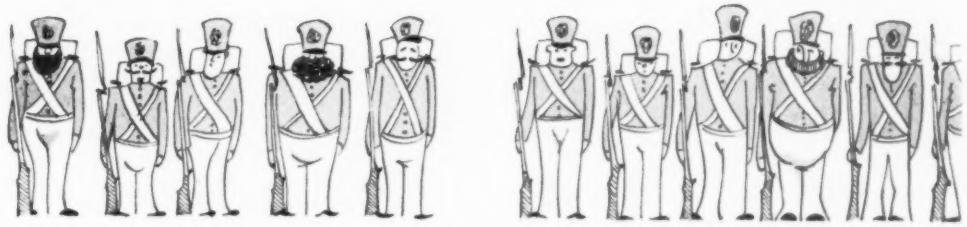
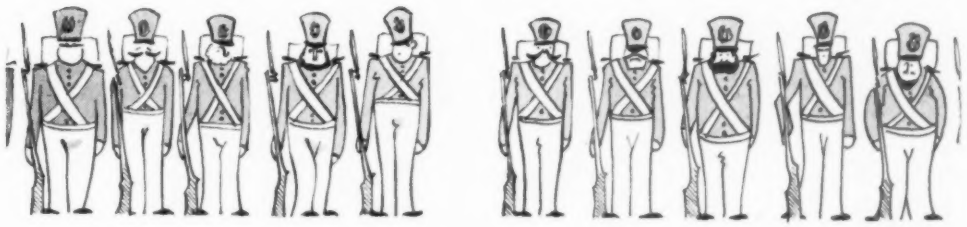


on parade for sizing



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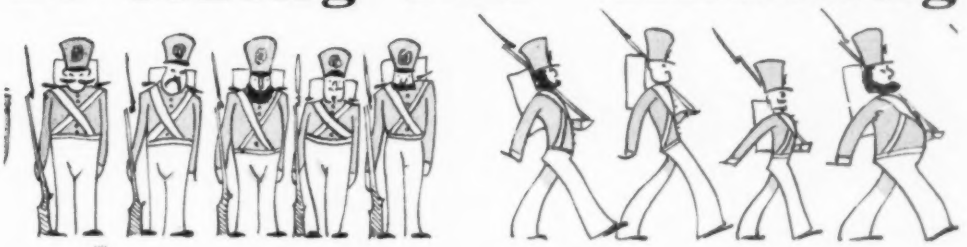

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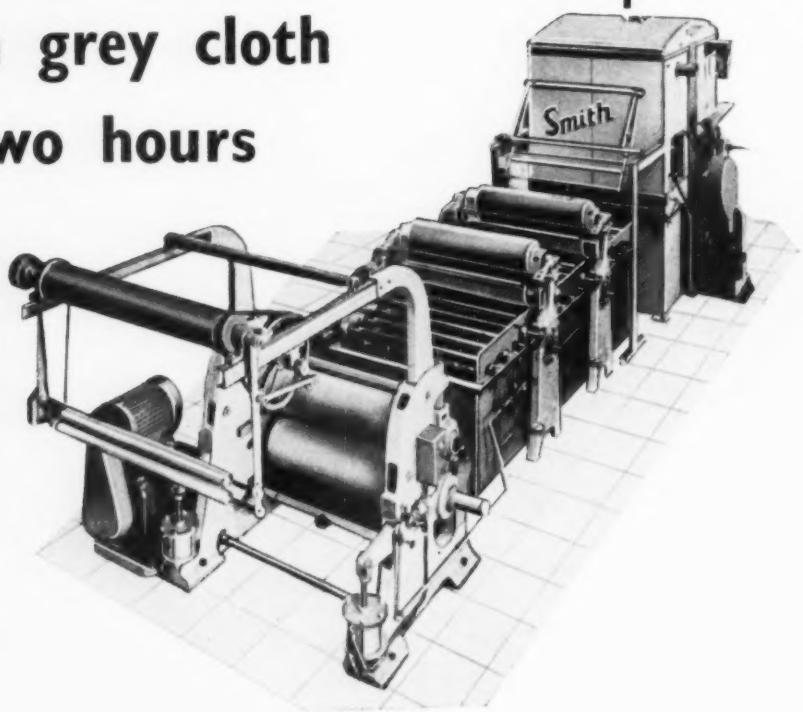

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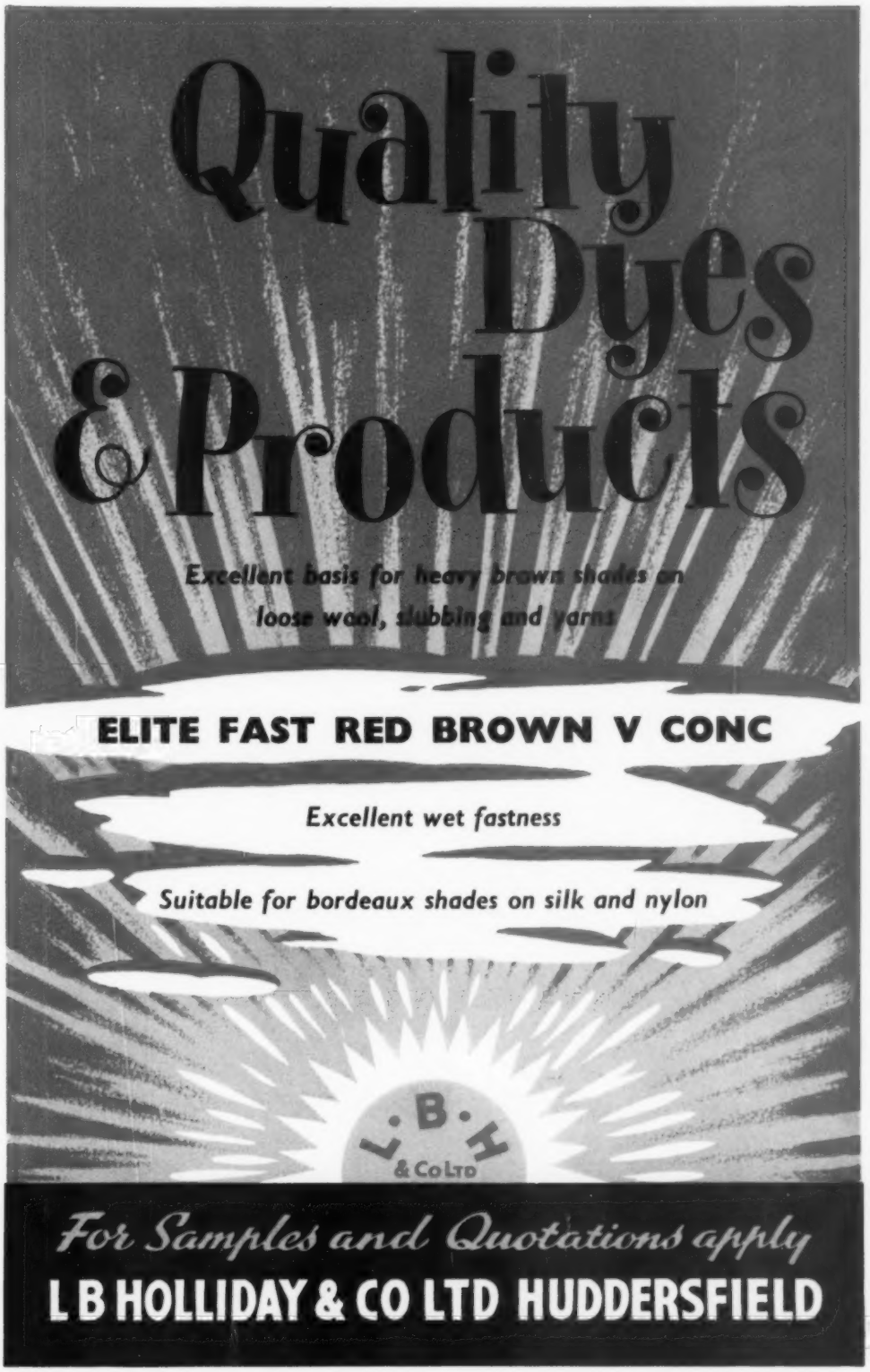
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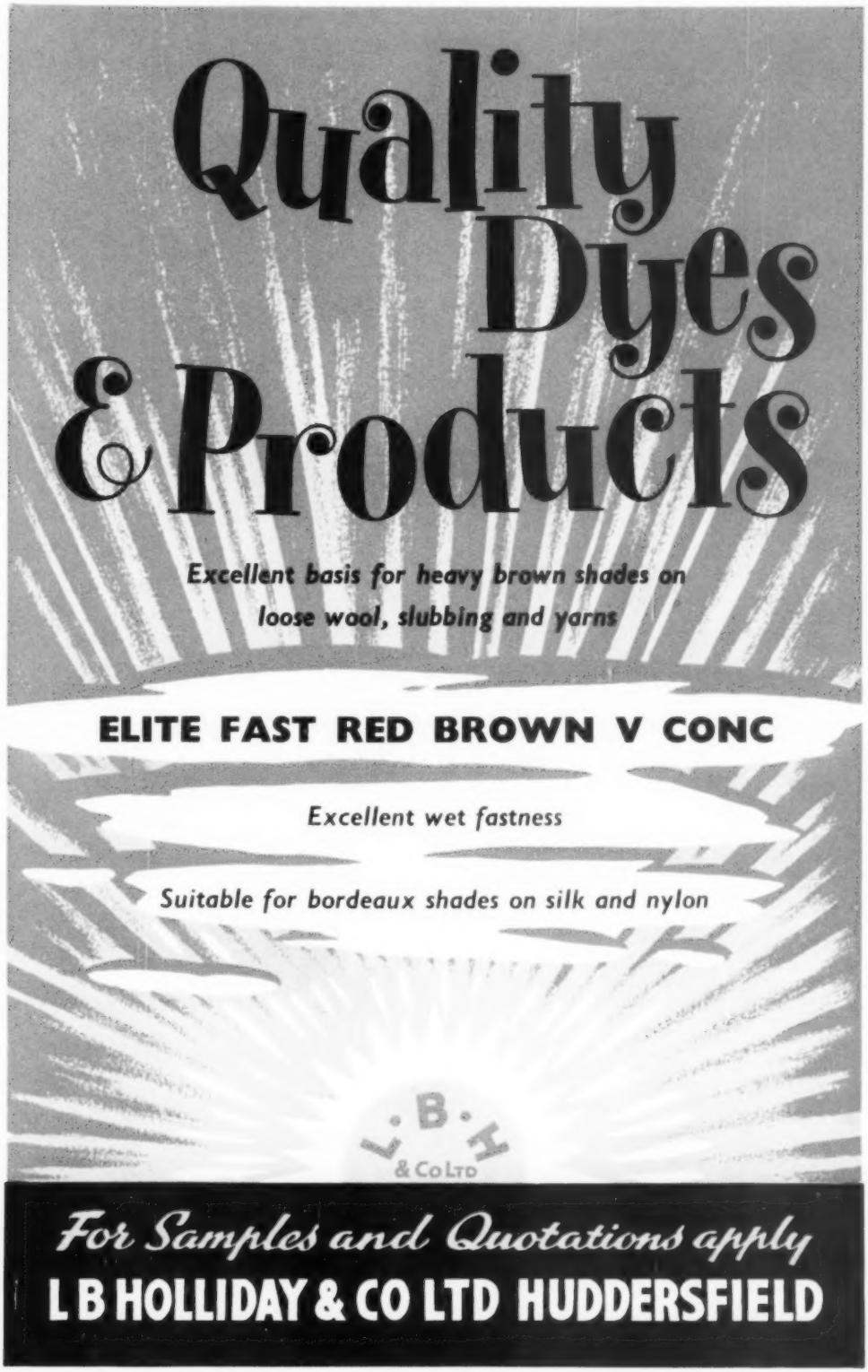
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Dyes
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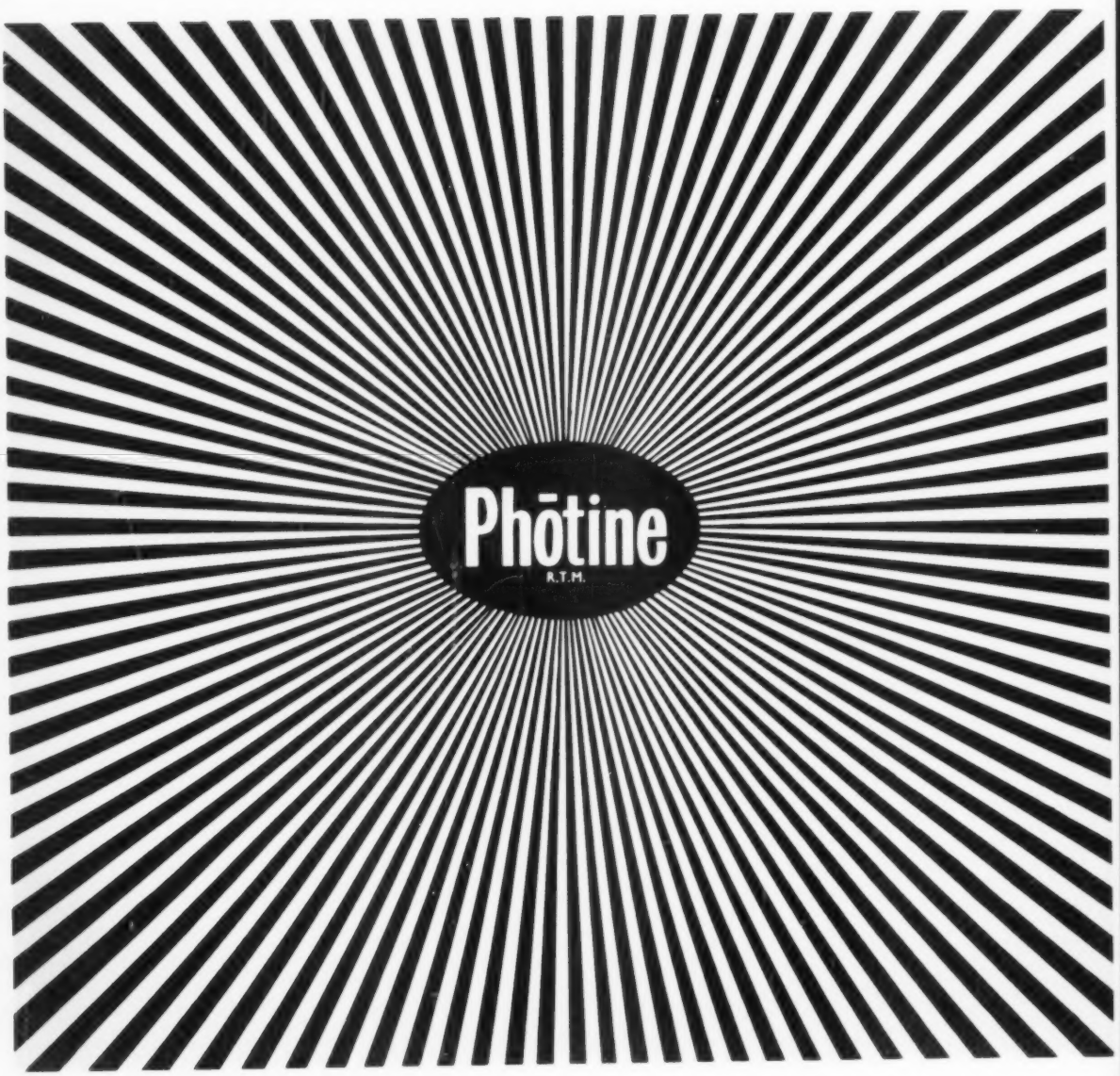

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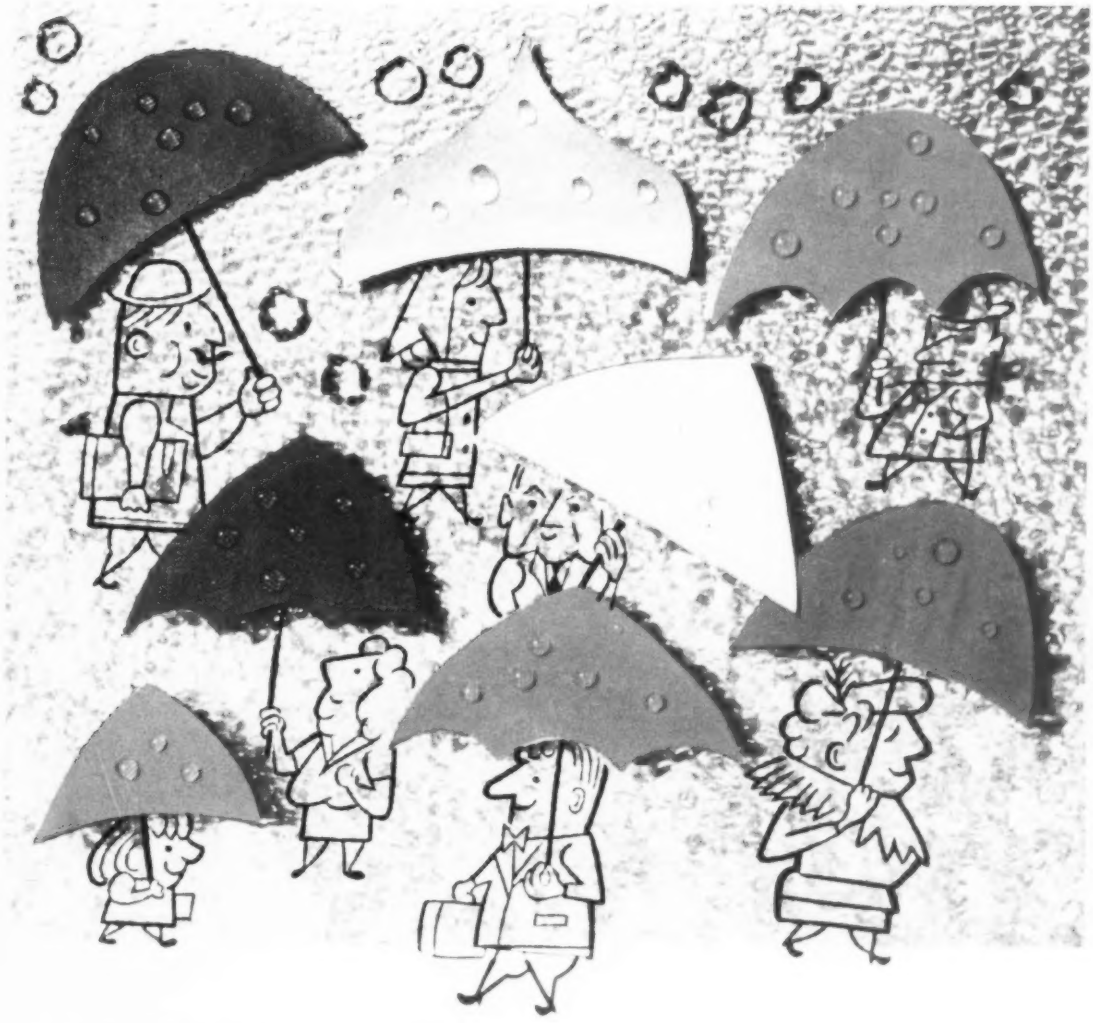

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with Isocyanates

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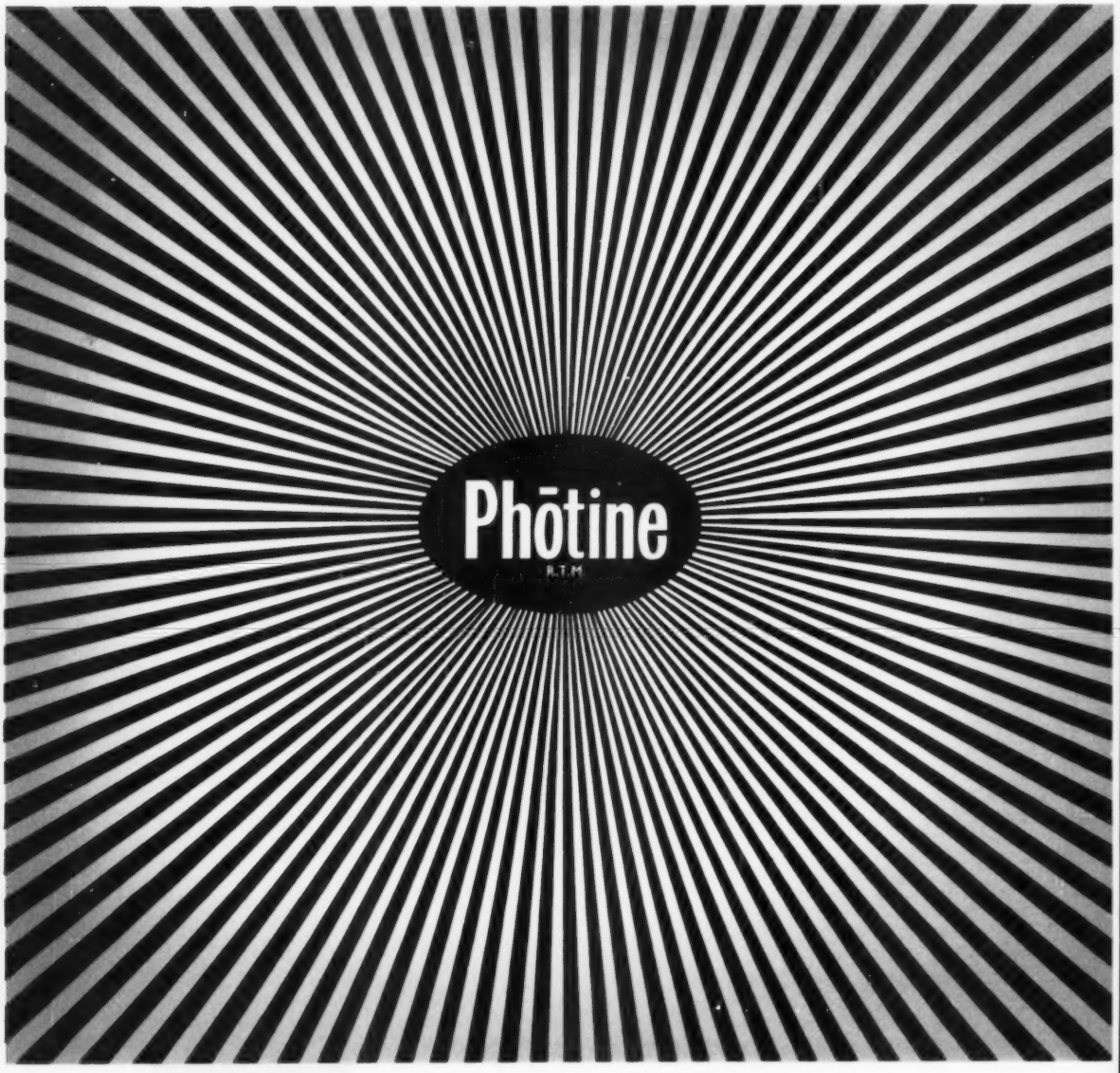

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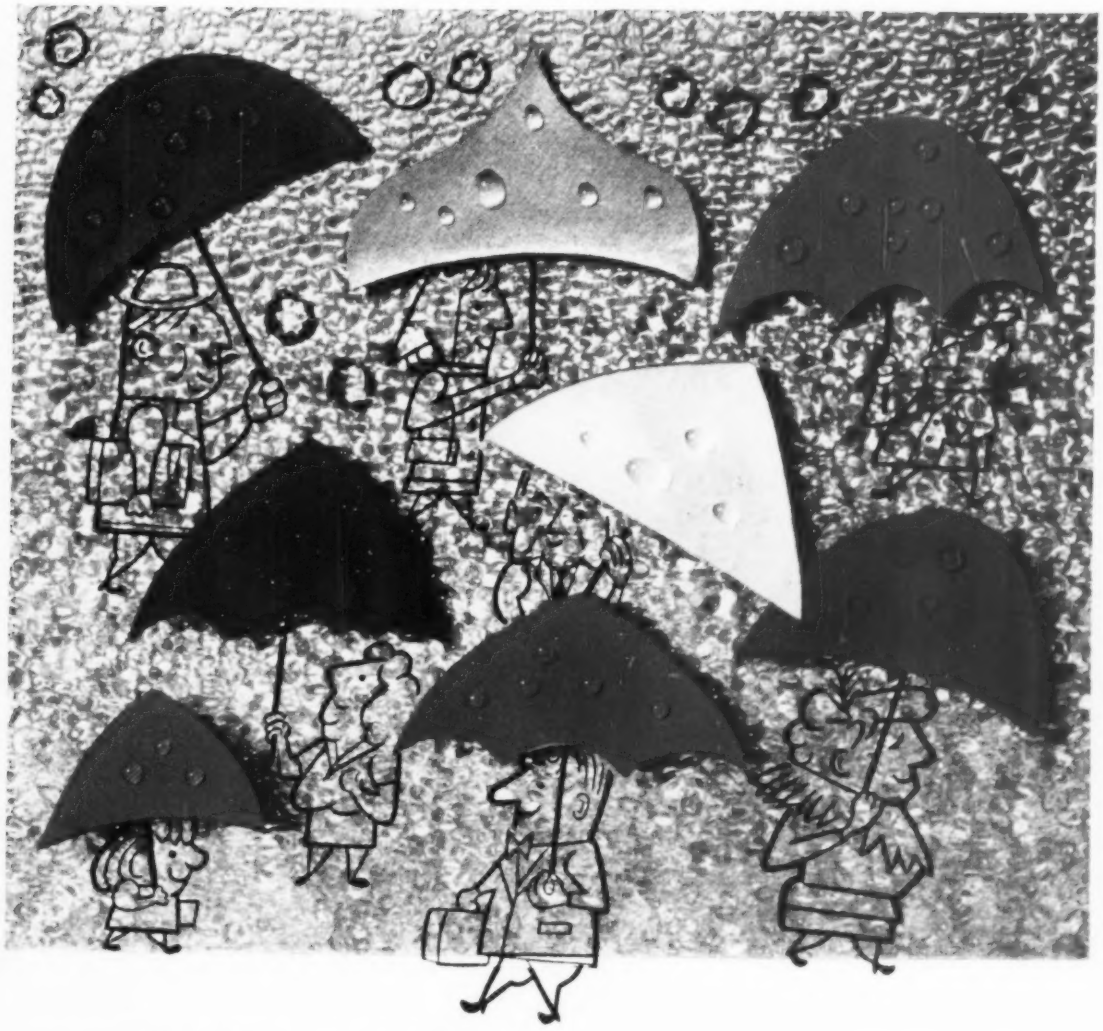

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Ask for particulars of these new proofing products **Daltolacs 10, 11 & 315, Suprasecs AC, C & DX**

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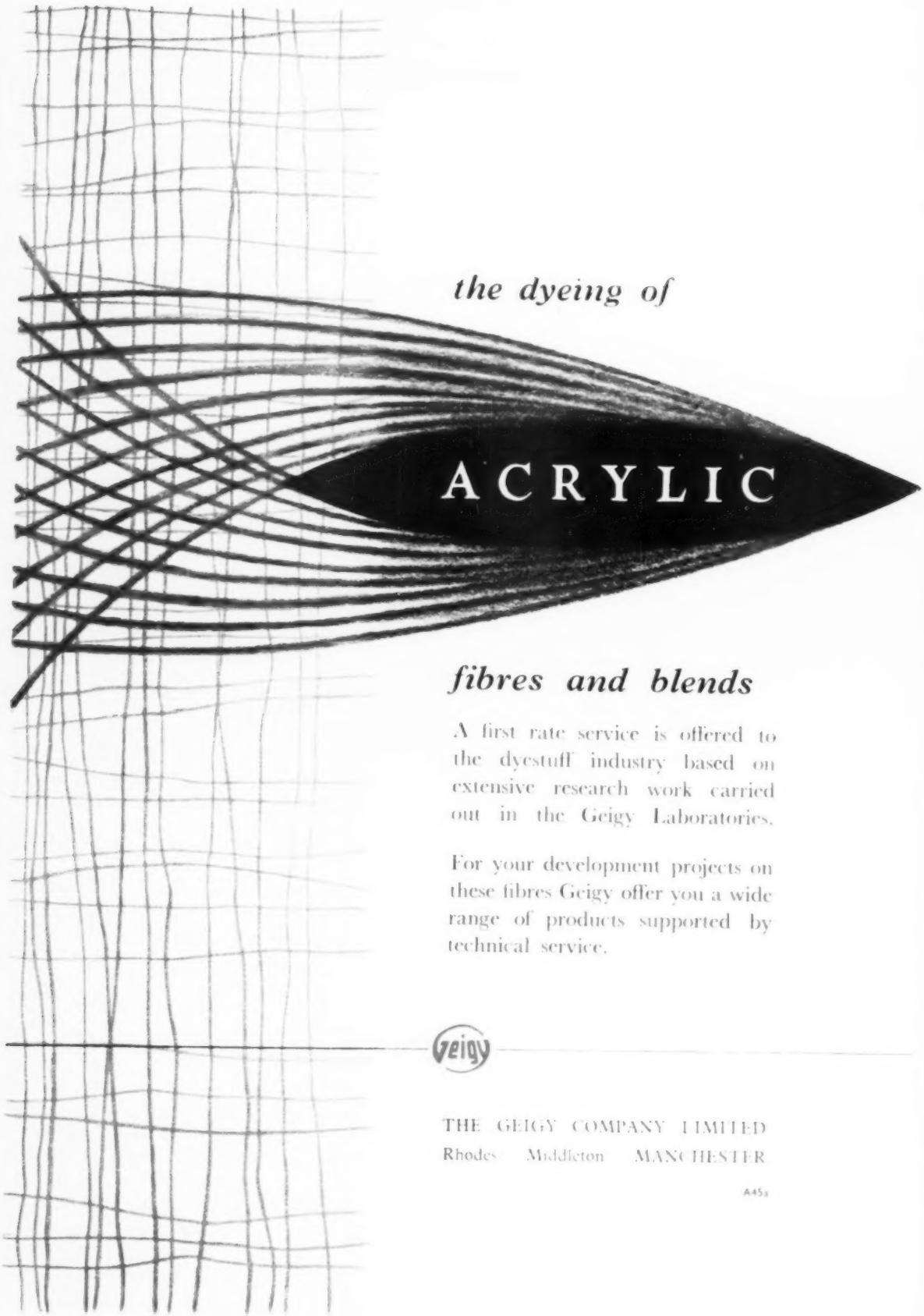

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A45a

so much in various ways for the Society, are no longer with us.

Then there was considerable difficulty in finding a method of contracting with the Americans owing to the constitution of the Society, but eventually Mr. Robert Conchar, for many years Honorary Solicitor to the Society, found a way round these difficulties, and on 18th January 1952, before the American Consul in Bradford, Mr. F. Scholefield as President, Mr. Jennison as Honorary Treasurer, and I as Honorary Secretary signed the Agreement on behalf of the Society.

In the year following I had to relinquish my office as Honorary Secretary owing to the increasing demands of the *Colour Index*, and thus you will appreciate that I cannot say much of all the work which has been put in since by those concerned with the general administration, but obviously the business negotiations and the arrangements for final printing and despatch must have been very onerous.

On the whole, the relationship of the Panel with its opposite number in the AATCC has been very good, though, as you would expect, we have at times had different viewpoints, and if I mention leather dyes I am sure that my colleagues will remember long and difficult discussions.

Let me conclude by paying tribute to my colleagues on the Panel, all of whom have worked splendidly. We have been a very happy team; sometimes there were divergent views, but differences never rankled, and over all these years we have got to know each other very well. I need not refer to the Panel members individually; many of you will know them, and in a few minutes, I understand, they will be presented to you.

In the final report of the Panel [p. 112] we made suggestions for keeping the *Colour Index* up to date, and I would suggest that the best tribute which could be paid to all who have taken any part in the production of the Second Edition is to see that this is done.

This year, Gray's *Anatomy* celebrates continuous publication and revision for 100 years, an event unique for a technical publication. Let us hope that the *Colour Index* may do this in the year 2024.

PRESENTATION OF AWARDS

The President of the Society then presented the various awards, as indicated in the list facing p. 110, to the accompaniment of loud acclamation.

"OUR GUESTS"

Mr. FRED SMITH (*a Past President of the Society*), in proposing this toast, said: This dinner and the awards are very small recompense for the time, thought, and work given over the past thirteen years (1945–1958), all without payment. I shudder to think what the cost would have been had it not been so, but that is the basis of much of the work done in the Society.

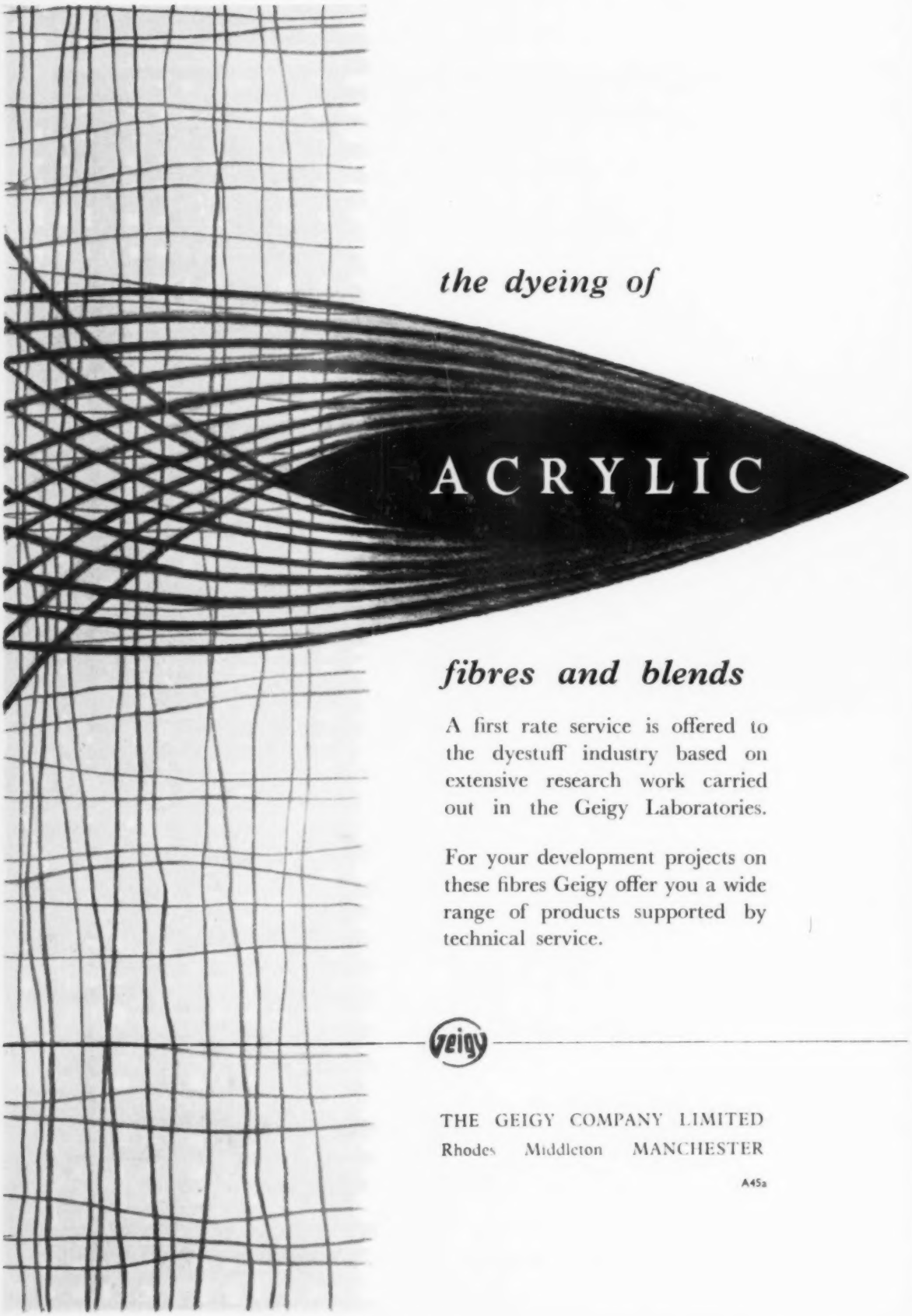
In the Foreword in Volume I of this new *Colour Index* the President of the Society and the President of the AATCC express thanks to the Editorial Panel and the various firms who collaborated.

I should be failing in my duty if I did not mention that there are others, not represented here tonight, many of whom took no actual part in the editing of this colossal work, who also should receive our thanks. I allude to those who had the courage to launch this costly project at a time when they wondered how it was possible to see it through at a cost of £45,000, that being the agreed maximum liability of this Society. Without mentioning any names, I know that it would be the wish of Council that they be included in this toast. Also, those who came to our assistance by making prepayments to assist with the financing of this work. The printers who, whilst receiving payment for their excellent work, must have had many harassing times; we thank them for their patience and toleration, and also the paid officials and staff, who must have done a lot of extra work.

This publication has caused many headaches, but all is well that ends well. We may consider that the first British colour index, dealing with synthetic dyes, a private effort in 1894 by A. G. Green, contained only 454 colouring matters; the second in 1904, by the same author and revised in 1908, contained 711 colouring matters, the First Edition of our *Colour Index*, started at the instigation of Ernest Hickson and published in 1924, contained 1316 colouring matters (in one volume); whilst the present Edition in four volumes contains approximately 3595 constitutions, plus thousands of other entries. What a boon the first *Colour Index* must have been to our British dyestuffs industry since 1924!

We thank you and are proud of everyone who has taken part.

Mr. JOHN FOSTER BEAVER, O.B.E., J.P. (*Chairman of the Wool Industries Research Association*), in response, recalled that twenty years ago, in that very room, he had replied to the toast to the guests at a dinner addressed by Dr. H. Dreyfus. On the present occasion he felt greatly honoured at being asked to reply on behalf of the guests at such a distinguished gathering, which included chairmen and managing directors of leading dyemaking and dye-using firms. He had been greatly impressed by what he had seen of the contents of the new *Colour Index*, and trusted that all the scientific achievements recorded in such publications would be put to proper use. On behalf of the guests he expressed grateful thanks to the Society for the good fare and good fellowship enjoyed that evening.



the dyeing of

ACRYLIC

fibres and blends

A first rate service is offered to the dyestuff industry based on extensive research work carried out in the Geigy Laboratories.

For your development projects on these fibres Geigy offer you a wide range of products supported by technical service.

Geigy

THE GEIGY COMPANY LIMITED
Rhodes Middleton MANCHESTER

A45a

so much in various ways for the Society, are no longer with us.

Then there was considerable difficulty in finding a method of contracting with the Americans owing to the constitution of the Society, but eventually Mr. Robert Conchar, for many years Honorary Solicitor to the Society, found a way round these difficulties, and on 18th January 1952, before the American Consul in Bradford, Mr. F. Scholefield as President, Mr. Jennison as Honorary Treasurer, and I as Honorary Secretary signed the Agreement on behalf of the Society.

In the year following I had to relinquish my office as Honorary Secretary owing to the increasing demands of the *Colour Index*, and thus you will appreciate that I cannot say much of all the work which has been put in since by those concerned with the general administration, but obviously the business negotiations and the arrangements for final printing and despatch must have been very onerous.

On the whole, the relationship of the Panel with its opposite number in the AATCC has been very good, though, as you would expect, we have at times had different viewpoints, and if I mention leather dyes I am sure that my colleagues will remember long and difficult discussions.

Let me conclude by paying tribute to my colleagues on the Panel, all of whom have worked splendidly. We have been a very happy team: sometimes there were divergent views, but differences never rankled, and over all these years we have got to know each other very well. I need not refer to the Panel members individually: many of you will know them, and in a few minutes, I understand, they will be presented to you.

In the final report of the Panel [p. 112] we made suggestions for keeping the *Colour Index* up to date, and I would suggest that the best tribute which could be paid to all who have taken any part in the production of the Second Edition is to see that this is done.

This year, Gray's *Anatomy* celebrates continuous publication and revision for 100 years, an event unique for a technical publication. Let us hope that the *Colour Index* may do this in the year 2024.

PRESENTATION OF AWARDS

The President of the Society then presented the various awards, as indicated in the list facing p. 110, to the accompaniment of loud acclamation.

"OUR GUESTS"

Mr. FRED SMITH (*a Past President of the Society*), in proposing this toast, said: This dinner and the awards are very small recompense for the time, thought, and work given over the past thirteen years (1945–1958), all without payment. I shudder to think what the cost would have been had it not been so, but that is the basis of much of the work done in the Society.

In the Foreword in Volume 1 of this new *Colour Index* the President of the Society and the President of the AATCC express thanks to the Editorial Panel and the various firms who collaborated.

I should be failing in my duty if I did not mention that there are others, not represented here tonight, many of whom took no actual part in the editing of this colossal work, who also should receive our thanks. I allude to those who had the courage to launch this costly project at a time when they wondered how it was possible to see it through at a cost of £45,000, that being the agreed maximum liability of this Society. Without mentioning any names, I know that it would be the wish of Council that they be included in this toast. Also, those who came to our assistance by making prepayments to assist with the financing of this work. The printers who, whilst receiving payment for their excellent work, must have had many harassing times: we thank them for their patience and toleration, and also the paid officials and staff, who must have done a lot of extra work.

This publication has caused many headaches, but all is well that ends well. We may consider that the first British colour index, dealing with synthetic dyes, a private effort in 1894 by A. G. Green, contained only 454 colouring matters; the second in 1904, by the same author and revised in 1908, contained 711 colouring matters; the First Edition of our *Colour Index*, started at the instigation of Ernest Hickson and published in 1924, contained 1316 colouring matters (in one volume); whilst the present Edition in four volumes contains approximately 3595 constitutions, plus thousands of other entries. What a boon the first *Colour Index* must have been to our British dyestuffs industry since 1924!

We thank you and are proud of everyone who has taken part.

Mr. JOHN FOSTER BEAVER, O.B.E., J.P. (*Chairman of the Wool Industries Research Association*), in response, recalled that twenty years ago, in that very room, he had replied to the toast to the guests at a dinner addressed by Dr. H. Dreyfus. On the present occasion he felt greatly honoured at being asked to reply on behalf of the guests at such a distinguished gathering, which included chairmen and managing directors of leading dyemaking and dye-using firms. He had been greatly impressed by what he had seen of the contents of the new *Colour Index*, and trusted that all the scientific achievements recorded in such publications would be put to proper use. On behalf of the guests he expressed grateful thanks to the Society for the good fare and good fellowship enjoyed that evening.

Report of the *Colour Index* Editorial Panel to the Council

With the publication of Volume 4 of the Second Edition of the *Colour Index* the formal task of the *Colour Index* Editorial Panel is completed. The Panel presents this short report of its activities to the Council of the Society, together with some suggestions which it hopes will be useful in the future.

The first meeting of the Panel was held on 5th October 1945, and at the 168th meeting held on 20th August 1958 this report was approved.

As stated in the foreword in Volume 1 by the Presidents of the Society and the AATCC, the present production has been a joint effort of the Society and the AATCC. The Second Edition differs from the First Edition in that—

1. In the First Edition only dyes of disclosed constitution were listed, whereas in the Second Edition all commercially homogeneous dyes are included, whether or not their constitutions have been disclosed
2. In the First Edition classification was solely by chemical constitution: whilst retaining this traditional classification in Part II of the Second Edition, Part I (which comprises Volumes 1 and 2) classifies the dyes by their conventional usage classes
3. In the Second Edition greater emphasis is laid on application and fastness properties.

The First Edition contained 1,316 dyes of different constitution. In the Second Edition 3,595 constitutions are given in Part II, and there are 4,612 entries in Part I. The commercial names index of the First Edition contained approx. 13,000 entries; that of the Second Edition approx. 31,500 entries. Dyemakers throughout the world were invited to supply information, and, apart from the U.S.S.R., the response has been virtually complete.

The members of the Panel are only too conscious of the time which has elapsed since this venture was first sponsored, but the task has proved to be much greater than was anticipated, and an enormous amount of detailed and tedious work has been necessary. Co-operation with the AATCC has led to unforeseen delays because, as would be expected, there have been differences in viewpoint between the two bodies, some of which have taken a considerable time to resolve as a result of the impossibility of meeting around a table. Throughout, our relations have been characterised by a spirit of goodwill and a desire to make the Second Edition of the *Colour Index* of the greatest possible use to industry.

As originally planned, it was thought that the *Colour Index* would run to five volumes, but in the event it has been possible to produce the work in four volumes with a consequent saving in paper, printing, and binding costs. The Panel has not been directly concerned with the financial background to the *Colour Index*, but naturally it took an active part in getting out the original estimates. It is pleasing to record that the final cost comes within the original budget. The provision of paper by the AATCC in the early stages was a help in that paper was not then available in this country. In passing, the co-operation of the Board of Trade in allowing entry of the paper into this country should be acknowledged, and also their generosity in providing the Panel with copies of the reports on the German dye- and pigment-making industries.

After the publication of the First Edition in 1924 and the Supplement in 1928, it was intended to keep the *Colour Index* up to date, but for various reasons this was never done, and this omission made the task of preparing the Second Edition so much greater.

In the Presidents' Foreword to the present edition it is noted that new material is continually coming forward and that it is the intention of both the Society and the Association to keep the *Colour Index* up to date by the issue of Supplementary Volumes at appropriate intervals. The Panel wishes to emphasise the importance of keeping the work of preparation in an active state, so that at any suitable time a Supplement may be issued.

An important current reason for keeping the *Colour Index* up to date is its value in registering dyes for import and export purposes.

The Panel submits herewith a scheme which deals with the preparation of supplements and the labelling of new dyes.

The Panel's relations with dyemakers has throughout been most cordial, and the numerous and difficult queries have always been treated with unfailing courtesy. Much of the credit for any success the *Colour Index* achieves is due to this co-operation from dyemakers.

The present members of the Panel have been privileged to see the completion of the work, but they wish to be the first to acknowledge the contribution made by former members, whose names may be seen in the complete list of members given in the Appendix.

In addition there are non-members of the Panel who have made considerable contributions to the compilation of the data, and in particular we would refer to Messrs. W. E. Wood and T. Wozniczka, who have compiled the sulphur dye sections for Parts I and II; Dr. F. F. Elsworth, who has helped to compile the acid dye section of Part I; Dr. E. J. Cross, who has revised the proofs of the azo dye section of Part II; Dr. B. C. Bhattacharya, who has helped with the natural organic colouring matters; and the Oil and Colour Chemists' Association, which has given assistance in the solvent dye and pigment sections of Part I.

The Panel cannot but place on record its high appreciation of the excellent and courteous co-operation given by Mr. W. H. Cady, the Co-ordinator of the AATCC *Colour Index* Editing Committee, during the whole of the thirteen years' work. The outstanding contributions of Dr. O. Stallmann and Dr. C. W. Maynard Jr. to the compilation of Part II are gratefully acknowledged.

Throughout the whole of the time the Panel has been well served by its clerical staff.

The work of the Panel has been greatly facilitated by the patience and wholehearted co-operation of the printers.

Lastly, the members of the Panel who have been permitted to take part in this work by their respective employers wish to record with gratitude the facilities granted to them to carry out the work, and the willingness with which such permission and facilities were given.

In submitting this report to Council, the members of the Panel express their appreciation of the trust which was placed in them in the compilation of a work of such size and cost. They believe that their task was a worthy one, and hope that their efforts will enhance the prestige of the Society.

JOHN BARRITT *Chairman*
H. BLACKSHAW *Vice-chairman and Co-ordinator*
C. O. CLARK *Co-ordinator*
W. BRADLEY
J. G. GRUNDY
R. J. HANNAY
H. H. HODGSON
M. LAPWORTH
A. THOMSON

August 1958

APPENDIX

Colour Index Editorial Panel,

J. Barritt B.Sc. A.R.C.S. A.R.I.C. F.S.D.C. *Wool Industries Research Association, Leeds*	Chairman 1949–58	1945–58
H. Blackshaw F.S.D.C. *Imperial Chemical Industries Ltd., Manchester*	Vice-chairman 1946–58, Co-ordinator 1947–58	1945–58
H. H. Bowen F.S.D.C. *Bradford Dyers' Association Ltd., Bradford*	Chairman 1945–49	1945–49
W. Bradley D.Sc. Ph.D. F.R.I.C. F.S.D.C. *University of Leeds*		1949–58
C. O. Clark F.T.I. F.S.D.C. *Bradford Dyers' Association Ltd., Bradford*	Co-ordinator 1955–58	1949–58
D. A. Clibbens B.Sc. Ph.D. Hon. F.T.I. *British Cotton Industry Research Association, Manchester*		1945–47
A. W. Doyle M.Sc.Tech. A.M.C.T. F.R.I.C. F.S.D.C. *College of Technology, Manchester*	Co-ordinator 1945–47	1945–47
J. G. Grundy F.S.D.C. *Clayton Dyestuffs Co. Ltd., Manchester*		1949–58
R. J. Hannay B.Sc. F.R.I.C. F.T.I. F.S.D.C. *Brotherton & Co. Ltd., Leeds*		1949–58
F. S. H. Head B.Sc. Ph.D. F.R.I.C. *British Cotton Industry Research Association, Manchester*		1947–57
J. S. Heaton M.Sc. Ph.D. *Calico Printers' Association Ltd., Manchester*		1945–49
H. H. Hodgson M.A. Ph.D. F.R.I.C. F.S.D.C.		1950–58
M. Lapworth B.Sc. A.R.C.S. A.R.I.C. F.S.D.C. *Imperial Chemical Industries Ltd., Manchester*		1946–58
*A. Shepherdson B.Sc. *Imperial Chemical Industries Ltd., Manchester*		1945–46
A. Thomson A.R.I.C. F.S.D.C. *Yorkshire Dyeware & Chemical Co. Ltd., Leeds*		1945–58

* Deceased 1946

Colour Index Editorial Board

The celebration dinner was a fitting conclusion to the work of the Editorial Panel in its preparation of the Second Edition of the *Colour Index*. Some members of the Panel have agreed to continue work on the *Colour Index* by supervising the preparation of the lists of Additions and Amendments which are to appear in the January, April, July, and October issues of the *Journal*.

The new *Colour Index* Editorial Board, with Mr. J. Barritt, O.B.E., as Chairman together with Messrs. C. O. Clark, J. G. Grundy, M. Lapworth, A. Thomson, and W. E. Wood, will in due course supervise the publication of the First Supplement.

New Books and Publications

The Dyeing and Finishing of Half Hose and other Footwear

By Arved Datyner. Pp. v + 23. Bradford: Society of Dyers and Colourists. 1958. Price, 7s. 6d.

In spite of its economic importance, hosiery usually receives very little attention in textbooks on dyeing and finishing, and it is very appropriate that this, the first of a series of monographs which the Society proposes to publish on various sections of the dyeing and finishing industry, should cover the processing of footwear. While it is primarily written for the benefit of students, the technologists of our Northern counties may be expected to find interesting reading in an account of the methods and techniques in use in the Midlands.

The author is to be congratulated upon the admirable manner in which he has carried out the intention expressed in his preface, namely to provide a monograph for the use of students in our technical colleges consisting of "expositions of practices . . . without fulsome detail and avoiding any processes which might be the subject of controversy . . .". Dr. Datyner has succeeded in presenting more basic information in the 23 pages of his text than is usually found in substantially longer chapters of conventional textbooks, yet without sacrificing an easily readable style.

The monograph is divided into four parts. Part I covers the dyeing and finishing of men's all-wool half hose, and describes in a concise manner the techniques and machines used in processing socks in general. Information is given on the methods commonly used in scouring, shrink-resisting, dyeing, drying, and trimming, with particular reference to the precautions necessary in handling this class of hosiery.

Part II deals with half hose made from yarns other than 100% wool and includes wool–nylon, wool–cotton, wool–viscose rayon, cotton, viscose rayon, nylon, and Terylene. The increasing importance of crimp nylon yarns in footwear is recognised, and mention is made of the special techniques required for the processing of this class of goods. Part III is a comparatively short section covering the bleaching of various qualities of socks classified according to fibre content, while Part IV briefly refers to footwear other than half hose. The processing of ladies' full-length hose has been intentionally excluded from the present monograph. An appendix includes a glossary of certain terms used in the hosiery industry, references to five patents covering shrink-resist processes, and a rather lengthy extract from the National Hosiery Manufacturers Federation *Code of Fair Trading* specifying shrinkage tests and tolerances.

There is one misconception to which attention must be drawn. In describing various shrink-resist patents in use on wool half hose the author refers to "Dylan" as a hypochlorite–permanganate process. *Dylan* is in fact a trade mark certifying that the goods concerned have received an approved shrink-resist treatment, and is not descriptive of any particular process.

The monograph is illustrated by a number of photographs and simple line diagrams of machines and equipment. The diagrams are well defined and annotated and will enable the student to obtain a clear idea of the mode of operation of the machines concerned.

Dr. Datyner has established an excellent precedent, and we look forward with interest to the publication of other contributions to this series.

H. W. PARTRIDGE

Perkin Centenary London
100 years of Synthetic Dyestuffs

Supplement No. 1 to *Tetrahedron*. Pp. xii + 136 + viii plates. London: Pergamon Press Ltd. 1958. Price, 42s. 0d.

The historic occasion of the Perkin Centenary Celebration has been admirably rounded off by the publication of this excellently produced and very readable book. The volume consists essentially of the lectures given in the Royal Institution two years ago, with a foreword by Sir Robert Robinson. The lectures included are *The Life and Work of Perkin* by Professor J. Read, *The Development of the Dyestuffs Industry* by Mr. Clifford Paine, *The Tinctorial Arts Today* by Mr. J. G. Evans, and *The Development of Organic Chemistry since Perkin's Discovery* by Sir Alexander Todd. The final chapter consists of the speeches made at the banquet by the Lord President of the Council (The Marquess of Salisbury, K.G., P.C.), by the then President of the Society of Dyers and Colourists (Mr. Clifford Paine), by the President of the Gesellschaft Deutscher Chemiker (Professor B. Helferich), and by the Prime Warden of the Worshipful Company of Dyers of the City of London (Mr. A. H. Brewin) and illustrates the wide appeal these Centenary celebrations made.

The book is stimulating in its approach to Perkin's discovery of Mauve and its repercussions. It emphasises the dual heritage left by Perkin—the first, commercial and scientific, leading to the vast industries based on organic chemistry and dyes, and the second a high standard of service by the dyemaker to the dye user which has grown out of Perkin's urge to find methods of applying the new dye he had found and to pass this information to the dyer.

In the pages of this book a third and in some ways the most interesting picture emerges, of Perkin the kindly man with his intense love of research and of music, and those of us who were privileged to know his son, Professor A. G. Perkin, will be more able to visualise the character of the man who, to quote from Professor Read—

> finding gold at the foot of the rainbow— that symbol of alchemy's elusive quest of the Philosopher's Stone— left it to his fellow beings to discover the transmuting tincture in his own heart.

Part of the proceeds from the sale of the volume will be donated to the Perkin Centenary Trust.

As the Society of Dyers and Colourists was a sponsoring body, members have the privilege of obtaining copies at the reduced price of 21s. 0d. each post free, by making application to the Society's office. A. THOMSON

Mercerisation

By Hans Rau. Pp. 57. Heidenheim/Brenz: Verlag Alfred Halscheidt. Price, DM 8.60.

This short book deals with the technique of mercerisation. Much of the material in it has little practical interest for the modern industry. The book includes a number of textile-finishing processes described in the patent literature of the last fifty years, many of which differ more or less widely from what is ordinarily described as technical mercerisation; few of these can ever have been used in bulk production. The subjects of most direct current interest are chainless piece-mercerising machines, and wetting agents for use in mercerising liquors. D. A. CLIBBENS

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University

Imperial Chemical Industries Ltd.

TECHNICAL INFORMATION LEAFLETS— *Dyehouse No. 428. Procion Dyestuffs: Application to Wool*— The skittery-dyeing properties and complete lack of migrating power of the dyes when covalently bound to the fibre still make it impossible for any general recommendations to be made for their application to wool. By dyeing in a weakly acid dyebath in presence of 2% Lubrol W and 1% Lissolamine A (50%) skitteriness may be reduced to a minimum, and details are given for dyeing loose wool, tops, and yarn in circulating-liquor machines and yarn in Hussong-type machines. By using the inherent skittery-dyeing properties of these dyes "ingrain" effects are obtainable. These are not affected by duration or rate of boiling and are thus more reproducible than those obtained with conventional dyes in presence of reserving agents. Fastness data for five Procion dyes on wool are given, and it is claimed that they give brighter dyeings of better fastness to light than any conventional wool dye. Brilliant Blue H7G is, perhaps, the most interesting, since it is superior in both dyeing and fastness properties to the direct blues of the same hue.

Dyehouse No. 441. Phototropism with Turquoise Blue Dyestuffs on Cellulosic Fibres— Dyeings of the bright turquoise blue direct and reactive dyes, e.g. Durazol Turquoise Blue GR and Procion Brilliant Blue H7G, may change to violet or reddish blue when irradiated in strong ultraviolet radiation or exposed for a prolonged period to intense sunlight, slow reversion to the original colour occurring when irradiation ceases. The Alcian Blues do not show this phototropism, the magnitude of which is increased when the goods are aftertreated with dye-fixing agents or given certain crease-resist finishes. Of the latter, melamine–formaldehyde finishes cause the least effect.

Dyehouse No. 442. Wet Fastness Properties of Procion Dyeings— Colour bleeding and staining of adjacent material may sometimes occur with Procion dyeings owing to either uncombined dye remaining in the goods after dyeing or to a small amount of dye being split off by hydrolysis of the dye–fibre linkage. It is concluded that incomplete washing-off is much more likely to be the cause of colour bleeding, and a more thorough washing-off will prevent its occurrence. The loosening of dye on goods stored under humid, acid conditions is extremely unlikely. Resin-finished goods hardly ever exhibit colour bleeding from either cause. Attention is drawn to the fact that Procion Brilliant Blue H7G is exceptional in that the unfixed dye has appreciable affinity for cellulose, and soaping must be correspondingly thorough. When deep dyeings are being produced and washing-off is likely to be inadequate, a Fixanol PN aftertreatment is beneficial to ensure good fastness to water. Fastness data are appended showing the effect of this aftertreatment on the light fastness of dyeings.

Dyehouse No. 445. Identification of Procion Dyestuffs on Cellulosic Fibres— A procedure is recommended in which the presence of azoic, vat, and sulphur dyes is eliminated by determining the solubility of the dye in chloroform and its behaviour with reducing agents. The presence of Procion dyes (except blues) is confirmed by their behaviour on reduction, diazotisation, and coupling. The Procion blues give strongly coloured reduction products, although the original colour returns much more slowly. They are distinguished from the vat dyes by being decolourised by cold weak sodium hypochlorite solutions.

Dyehouse No. 455. Procion Dyestuffs: Selection of Resisting Agents for the Production of Illuminated Resists under Aniline Black— Magnesium oxide, carbonate, and hydroxide, zinc oxide and carbonate, and titanium oxide have been examined for suitability for the production of resists under Aniline Black. Magnesium oxides and hydroxides give the best resists in terms of clarity and colour yield. Satisfactory results may be obtained with the other compounds (except titanium dioxide) under certain conditions. A test specification for magnesium oxide is given and a tabular survey is given of the properties and behaviour in resist printing, of nine different samples of magnesium compounds.

Dyehouse No. 457. Coverage of Dead or Immature Cotton— Notes are given on the general ability of the various classes of dyes to cover dead cotton. The members of the direct cotton, vat, solubilised vat, and reactive types have been classified into three groups— (i) suitable, (ii) moderately suitable, and (iii) unsuitable— on the basis of comparisons by batchwise dyeing methods using a 30:1 liquor ratio, which probably represents the most adverse conditions (encountered in winch dyeing).

DIRECT DYES ON VISCOSE RAYON. VOLUMES 1, 2, AND 3— These three cards contain dyeings in three depths (two for blacks) on continuous-filament viscose rayon cloth of 111 direct dyes. In addition, Volume 3 contains dyeings in two depths of a further 19 dyes suitable for diazotisation and development on the fibre; these include the Chlorazol Diazo range specially designed for such aftertreatment as well as a number of dyes which are also of interest for direct dyeing or for use in conjunction with other aftertreatments. Each dye is allotted a separate page, and the now familiar loose-leaf format has been adopted. Fastness data are appended for the straight dyeing and such aftertreated dyeings as are recommended in each case. Volume 1 contains 32 pp. of text describing the dyeing behaviour of the dyes and methods of aftertreatment together with notes on the treatment of viscose rayon prior to dyeing, accelerated fading, the effect of sequestering agents, the dyeing of mixture materials, the selection of dyes for use on materials to be crease-resisted, and fastness properties and their assessment.

PROCION DYESTUFFS IN TEXTILE PRINTING. VOLUME 3— This third binder has been issued to accommodate the loose-leaf sheets describing individual dyes which can no longer be contained in Volumes 1 and 2, since a revised and enlarged introductory text (52 pp.) has now been issued to replace the text (22 pp.) originally issued with Volume 1. In addition, the new text supersedes all the

information dealing with Procion dyes in printing contained in *Technical Information Leaflets* (*Dyehouse*) from No. 309 to No. 404 inclusive.

WOOL SLUBBING. ACID MILLING DYESTUFFS FAST TO LIGHT AND MILLING. VOLUME 3.— The introduction of further dyes has necessitated the issue of a third binder to accommodate their data sheets, Carbolan Rubine 2B being the first dye to be included in it.

ALCIAN "X" DYESTUFFS IN TEXTILE PRINTING— This card contains prints on cotton of seven Alcian dyes, the range now comprising Alcian Yellow GX, Blues 8GX, 7GX, 5GX, and 2GX, and Greens 3BX and 2GX. A number of patterns from bulk printings are also included to illustrate the use of the dyes alongside vat dyes, solubilised vat dyes, and Soloxan Black, on Brenthol prepares and as coloured resists under Brentamine Fast Blue VB Salt on Brenthol AS prepare. The 29 pages of text include all the information contained in the *Technical Information Leaflets* (*Dyehouse*) up to and including No. 355.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I— PLANT; MACHINERY; BUILDINGS

Fleissner Continuous Dyeing and Drying Plant

H. Steglich

Melliand Textilber., **39**, 1032–1034 (Sept. 1958)

Rapid compact efficient multipurpose machinery specially suitable for loose fibres is described. S.M.J.

PATENTS

Carbon Black Furnace

Phillips Petroleum Co. *USP* 2,839,825

Controlling Tension in Web Material

Firestone Tire & Rubber Co. *BP* 805,017

During the heat stretching of nylon tyre fabric accurate control of the tension is obtained by passing the fabric through one or more groups of rolls. Each group comprises several rolls associated in pairs, each pair being driven at a speed different from that of the preceding pair so that change in tension is distributed throughout each group of rolls. This minimises roll wear and ensures that the fabric is not damaged during stretching. C.O.C.

Paper Drying Machine

G. S. Brendel and H. J. Voorhees *USP* 2,828,552

Air is supplied to the moist sheet by sets of accurately spaced tubes which are arranged transversely to the web and close to the upper portion of the surface of the drum, so as to disrupt the vapour and allow free escape of vapour-entrained air between the tubes, thus preventing re-use of wet air. The sets of tubes are arranged circumferentially in sequence, a partition dividing the tubes toward the delivery side of the drum, where the sheet is drier, from the feeding side. Hot dry air is supplied first to the tubes near the delivery zone and recycled to the tubes of the first sets. R.A.

Preventing the Escape of Vapour from Photogravure or other Printing Apparatus

Schnellpressenfabrik Frankenthal Albert & Cie. *BP* 805,579

Washing, Dry Cleaning and Garment Dyeing Machines

Isaac Braithwaite & Son, Engineers *BP* 805,624

Modification of *BP* 640,974 to enable the machine to perform the additional function of promoting circulation of liquid within the machine. C.O.C.

Printing or Decorating in Two or More Colours on Curved Ceramics, Glass, Plastics, etc. (IX p. 132)

II— WATER AND EFFLUENTS

PATENTS

Removal of Residual Active Chlorine from Water

Farbenfabrik Wolfen *BP* 805,659

Aromatic polyamine formaldehyde resins having an open-lattice structure of pore diameter 20–50 A. rapidly and completely remove residual chlorine, present as hypochlorite or hypochlorous acid, from water. C.O.C.

Clarifying Water

Dow Chemical Co. *BP* 805,526

Addition of a water-soluble acrylamide polymer to the coagulant results in formation of a rapid settling floc and reduces the amount of coagulant needed. C.O.C.

III— CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

New Solvent for Cellulose using the System Iron-Tartaric Acid-Alkali Hydroxide

G. Jayme and W. Bergmann

Naturwissenschaften, **43**, 300 (1956): *Chem. Abs.*, **52**, 17699 (25 Oct. 1958)

Aqueous solutions of ferritartrate, $Na(C_4H_2O_6)Fe$, (75–350 g./litre) containing excess free NaOH (0·5–3·0 *N*) dissolve native cellulose to a dark brown solution. Addition of Na tartrate markedly increases the solubility of cellulose in this solvent. K ferritartrate behaves similarly in excess KOH. Viscosity measurements indicate that O attacks the solutions of cellulose obtained with these solvents. C.O.C.

Synthesis of Tetrakistriethylsiloxytitanium and Tetrakistriethylsiloxytin

K. A. Andrianov and A. A. Zhdanov

Izvestiya Akad. Nauk S.S.S.R., otdel khim. nauk, 779–780 (June 1958)

The above compounds are synthesised by the exchange reaction of $(C_2H_5)_3SiONa$ with $TiCl_4$, or $SnCl_4$ respectively. G.J.K.

PATENTS

Asymmetrical Ester Salts of Thiophosphoric Acid

Ciba *BP* 805,931

Compounds of formula—

$$\begin{matrix} R^1O \\ R^2O \end{matrix}\!\!>\!PO{\cdot}S{\cdot}M$$

(R^1 and R^2 = different alcohol radicals; M = cation), e.g. the ammonium salt of dodecylethylthiophosphoric acid, are readily soluble or dispersible in water. They have wide uses, e.g. suitably substituted compounds can be used as detergents, wetting agents in mercerising lye, emulsifying agents, etc. C.O.C.

Antistatic Agent

Montecatini Società Generale per l'Industria Mineraria e Chimica *BP* 804,964

A mixture of a polyglycol, an ethylene oxide-glycerol condensate and an ethylene oxide-lauryl alcohol condensate is readily emulsifiable in water, and has good wetting power, low viscosity and excellent antistatic properties. C.O.C.

Size for Hydrophobic Fibres

Rohm & Haas Co. *BP* 804,952

A mixture of (1) a water-dispersible ammonium or alkali metal salt of a copolymer of 5–20% of acrylic or methacrylic acid with 95–80% of at least one acrylate or methacrylate of aliphatic alcohols of 1–18 C, at least half of this ester component being acrylates of primary or secondary acyclic alcohols of 1–18 C, methacrylates of primary or secondary acyclic alcohols of 6–18 C, the copolymer having a Knoop hardness of $\geqslant$ 7, and (2) a hydrophilic polymeric colloid, is a good size for nylon, etc. and is readily removed during normal dyeing and finishing. Thus an aqueous solution of a water-dispersible ethylene oxide-modified starch, sulphonated tallow and the ammonium salt of a copolymer of 20 mole % acrylic acid and 80 mole % ethyl acrylate having a Knoop hardness of 1, is an excellent size for nylon warps and is readily removed during scouring. C.O.C.

Antistatic Composition
Union Carbide Corpn. *BP* 805,549

An antistatic composition for application to hydrophobic synthetic resinous materials consists of an aqueous mixture of a polymerised polyethylene glycol acrylic ester and 1–16% on weight of the ester of an inorganic compound containing boron, e.g. borax. C.O.C.

Hair Waving Compositions containing Secondary Amino Aliphatic Alcohol Dithiocarbamates
Van Ameringen-Haebler *USP* 2,841,530

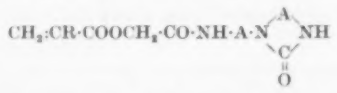
The product obtained by reacting together a secondary amino aliphatic alcohol, CS_2 and a halogenated lower aliphatic monocarboxylic acid or carboxylate or glycerol monochlorohydrin, e.g. the compound CH_3·CHOH·CH_2·N·CS(S·CH_2COOH)·CH_2·CHOH·CH_3, works rapidly without any deleterious effect and without imparting any odour to the hair. C.O.C.

Increasing the Solubility of Water-soluble Mothproofing Agents
FBy *BP* 805,774

Addition to mothproofing agents based on triphenylmethane of arylurea or diphenyl ether derivatives of a compound of formula X·CO·NR^1R^2 (X = H, Alk of 1–5 C, an aryl-O-, aryl-S- or aryl-NH-group; R^1 and R^2 = H or Alk), much increases their solubility in water. Thus addition of its own weight of monophenylurea or benzamide to 2:2′-dihydroxy-3:5:3′:5′:4″-pentachlorotriphenylmethane-2″-sulphonic acid renders it readily soluble to a clear solution by pouring over it 4–5 times its weight of boiling water; the solution can be diluted with water to any extent. C.O.C.

N-Acryloxyacetamidoalkyl-*NN*′-alkyleneureas
Rohm & Haas Co. *BP* 805,380

Compounds useful as plasticisers for nylon, vinyl resins, etc. to which they also impart increase in regain and consequently reduction in the tendency to accumulate static electricity, are of formula—



(R = H or CH_3; A = alkylene of 2–3 C), e.g. *N*-[β-(α-acryloxyacetamido)ethyl]-*NN*′-ethyleneurea. They are also used as modifiers for aminoplast resin-forming condensates. They react at the double bond with various compounds, thus addition of long chain amides or mercaptans yields water-repellent, softening and lubricating agents for textiles, leather, and paper. C.O.C.

Proteolytic Enzyme
American Cyanamid Co. *BP* 804,608

A fungus of a species of the order *Entomophthorales* is fermented in an aqueous nutrient medium containing assimilable sources of C, N and inorganic salts preferably including trace elements, under aerobic conditions and the resulting proteolytic enzyme recovered by absorption from the medium. It may be used for desizing, bating of leather, removal of egg albumen spots from garments, etc. It is stable between pH 4·0 to about 11·0. C.O.C.

Thickening Agents
Deutsche Gold- und Silber-Scheideanstalt vormals Roessler *BP* 805,166

The products obtained by treating an aqueous suspension of a natural substance containing SiO_2 in bonded or free form under pressure at 150–280°c. with an alkaline earth metal hydroxide, the ratio of solid to liquid in the suspension being about 1:6, preferably 1:10, are of irreversibly increased bulk, their s.g. being reduced to ≯ 10% of that of the original material. They are very efficient thickening agents, surprisingly pronounced effects being obtained by the use of small quantities. C.O.C.

Water-repellent Finish by use of Aqueous Solutions of Alkali Metal Siliconates
Monsanto Chemicals *BP* 804,990

Aqueous solutions of alkali metal siliconates containing as catalyst the reaction product of an amine with $TiCl_4$ or a titanate ester are used to impart a water-repellent finish to textiles and other fibrous or porous materials. C.O.C.

Titanate Esters of Polyhydric Alcohols as Catalysts for use in Aqueous Emulsions of Silicone Resins
Monsanto Chemicals *BP* 804,989

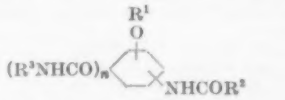
Aqueous emulsions of silicone resins can be catalysed with the titanates of polyhydric alcohols. These titanates are water-soluble and stable enough to water. Thus $TiCl_4$ (190 g.) and glycerol (224) were heated at 115°c. until a water-soluble liquid containing 14% Ti was formed. To this liquid (343 g.) was added triethanolamine (447) and the mixture brought to 170°c. The glyceryl titanate catalyst obtained was a homogeneous liquid which formed no precipitate on dilution with water. C.O.C.

Fillers and Delustring Agents for Rubber, Plastics, Paper, Fibres or Leather
Deutsche Gold- und Silber-Scheideanstalt vormals Roessler *BP* 804,834

The silicates obtained by treating an aqueous suspension of a natural substance containing SiO_2 in bonded or particularly in free form, e.g. bentonite, with an alkaline earth metal hydroxide under pressure at 150–280°c. have an irreversibly increased bulk, their s.g. being ≯ 10% of that of the bentonite. They have wide uses as fillers and delustring agents. C.O.C.

Mordants for Basic Dyes in Organic Colloid Layers
Kodak *BP* 804,661

Compounds of formula—



(n = 0 or 1; R^1 = monocyclic Ar of the benzene series containg Alk of > 3 C; R^2 = an alkali metal or ammonium monocyclic sulphoaryl or carboxyaryl group of the benzene series; R^3 = subst. or unsubst. monocyclic Ar of the benzene series), e.g. 5-[4-(2:4-di-*tert.*-amylphenoxy)-3-(phenylcarbamyl)phenylcarbamyl]-1:3-benzenedisulphonyl chloride, when used as mordants for basic dyes, greatly improve the stability of the dyes in organic colloid layers, e.g. in gelatin, polyvinyl alcohol, and hydrolysed cellulose ester. They do not interfere with the bleaching properties of the dyes in photographic processes. C.O.C.

Flame-resisting Agents for Cellulose
Gy *BP* 804,745

Modification of *BP* 790,663 (J.S.D.C., **74**, 491 (June 1958)) by replacing the amides of orthophosphoric acid by those of polyphosphoric acids containing < 4 P atoms and having > 3 hydroxyl replaced by monoalkyl amino groups the other hydroxyls being replaced by mono- or dialkylamino or alkoxy groups, the organic radicals containing < 5 C. Thus mercerised cotton muslin impregnated with its own weight of an aqueous solution containing 1:1′:3:3′-tetrakismonomethylamino-2-dimethylamino triphosphoric acid (16%) and trimethylol melamine (16%), dried and baked for 20 min. at 120°c. is flameproof, even after being washed with soap, when tested with the AATCC Flammability Tester. C.O.C.

Urea Glutarate as Printing Assistant
ICI *BP* 805,507

Urea glutarate is a valuable assistant in the process described in *BP* 793,566 (J.S.D.C., **74**, 564 (July 1958)) for the printing of dyes containing an *iso*thiouronium salt group attached through methylene linkage to an aromatic nucleus. If desired they can be mixed with ≮ 50% by weight of urea salts of other aliphatic dicarboxylic acids. C.O.C.

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

p-Hydroxyphenetole
M. Khalifa and A. A. Abo-Ouf *J.C.S.*, 3740 (Oct. 1958)

Lowering the electron-density on the azo-group should favour its reduction to the hydrazo-stage by zinc, and, in conformity with this view, *p*-azoxyphenetole is now found to reduce by zinc in ethanolic KOH to *p*-hydrazophenetole approx. twice as fast as *p*-azophenetole. H.H.H.

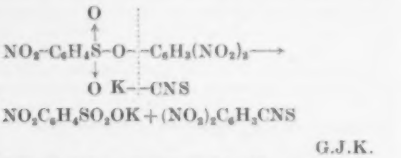
Peter Griess— A Hundred Years of his Diazo Reaction
D. H. Wilcox
Amer. Dyestuff Rep., **47**, 539–546 and 562 (11 Aug. 1958)

Survey of U.S. Dye Manufacture and Imports
Anon.
Chem. Eng. News, 34 (10 Nov. 1958)

Total U.S. dye production in 1957 was 143 million lb.— about equal to that in 1954 and the smallest since 1949. In 1946 cost of manufacture of dyes averaged 65 c. per lb., in 1957 it was $1·29. This increase in cost of manufacture is only partly explained by price increase of 30%, the greater part is caused by the use of more expensive dyes especially for dyeing synthetic fibres. Indigo (351,200 lb.) was the largest single item in 1957 dye imports, second largest being a fluorescent dye, Shirosol, of which 330,690 lb. were imported. Competition from European makers both in the U.S. and other countries has caused the U.S. production of synthetic indigo 20% paste to fall from 26·1 million lb. in 1950 to only 8·1 million lb. in 1957. According to a recent study made by Stanford Research Institute the annual growth of the U.S. dye industry was only 1·6% during the period 1929–1955, in contrast production of synthetic fibres had an annual increase of 36·1% during the period 1940–1954. C.O.C.

Reactions of Esters of Aromatic Sulphonic Acids. VII— Arylating Properties of Dinitrophenylbenzenesulphonates
R. V. Vizgert and Ya. P. Berkman
Zhurnal obshcheĭ khimii, **28**, 2119–2122 (Aug. 1958)

The esters formed from the reaction of 2:4-dinitrophenol with benzene-, *p*-chlorobenzene- and *o*-nitrobenzenesulphonic acid are reacted with ammonia, aromatic amines, KCNS and KI respectively. The corresponding substituted dinitrodiphenylamines and 2:4-dinitrothiocyanate-benzene are produced and a carbon–oxygen fission, as exemplified by the following equation, is indicated—



$$NO_2\text{-}C_6H_4SO\text{-}O\text{-}C_6H_3(NO_2)_2 \longrightarrow NO_2C_6H_4SO_2OK + (NO_2)_2C_6H_3CNS$$

G.J.K.

Compounds Containing the Epoxide Grouping
XXII— Interaction of the Esters of Glycidic Acids with α- and β-Naphthylamine
V. F. Martynov and N. P. Sukhinin
Zhurnal obshcheĭ khimii, **28**, 2075–2081 (Aug. 1958)

XXIII— Interaction of the Ethyl Esters of β-Methyl-β-butyl-, and β-Methyl-β-phenylglycidic Acids with Aniline
V. F. Martynov and Ya. A. Kastron
Ibid., 2082–2085

Preparation of Some New Substituted Naphthylamines and Naphthidines
R. Belcher, S. J. Lyle, and W. I. Stephen
J.C.S., 3243–3248 (Oct. 1958)

The Fries and Lohmann oxidation of 2-methyl-1-naphthylamine to 3:3′-dimethylnaphthidine (cf. *Ber.*, **54**, 2912 (1921)) is applied to the oxidation of 2-subst. 1-naphthylamines (including some new 2-*n*- and 2-*iso*propyl-1-naphthylamines), and is shown to be a general reaction for the preparation of 3:3′-disubst. naphthidines. A mechanism is suggested for the reaction and some limitations are noted. H.H.H.

Colour and Substantivity of Direct Azo Dyes— Derivatives of Diphenylamine
B. M. Krasovitskiĭ, Z. N. Tarakhno, and N. F. Levchenko
Ukrain. khim. zhur., **24**, 358–363 (1958)

The colour and substantivity towards cotton of mono- and dis-azo dyes from diphenylmethane, diphenyl oxide, diphenyl sulphide, diphenylamine, and *N*-methyldiphenylamine are investigated. It is claimed that the colour of substantive disazo dyes from 4:4′-diamino-diphenylamine cannot be interpreted solely by present-day theories. The authors suggest an additional factor to explain colour deepening in these compounds. G.J.K.

Reduction of *N*-Phenylhydroxylamine with Lithium Aluminium Hydride
M. L. Burstall and M. S. Gibson
J.C.S., 3745–3746 (Oct. 1958)

The reductions of nitrobenzene, nitrosobenzene, and azoxybenzene with lithium aluminium hydride to azobenzene with absence of aniline, has generally been attributed to the formation and subsequent reactions of *N*-phenylhydroxylamine (I). It is now shown that the reduction of I by the same reagent gives azobenzene and aniline in nearly equimol. amounts. The absence of aniline from the reduction products of nitrobenzene and nitrosobenzene indicates that I is not formed as a reaction intermediate in either case; if formed transiently and then undergoes preferential reaction, a mechanism must be postulated for the complete removal of the aniline so formed, e.g. condensation with nitrosobenzene to give azoxybenzene. The reduction of I can also be explained on the basis of its disproportionation to aniline and azoxybenzene before reduction. H.H.H.

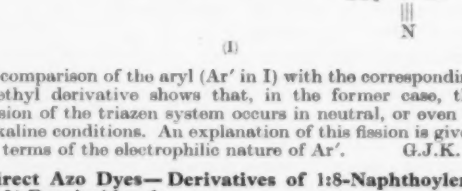
Reaction of Diazo Compounds with Sulphamic Acid and its Derivatives
D. Z. Zavel'skiĭ and L. A. Lishnevskaya

XI— Colour Phenomena of Some Diazonium and Heterocyclic Ammonium Salts
Zhurnal obshcheĭ khimii, **28**, 2560–2567 (Sept. 1958)

The diazonium bromide, iodide, and thiocyanate, assigned by Hantzsch the syn diazo structure, are stated to be true diazonium salts. These coloured compounds are shown to have similar properties to aryl sulphaminates and acyl amino sulphonates of diazonium compounds. The phenomenon of colour in these compounds and also in the ammonium salts of the heterocyclic series (pyridinium, quinolinium, etc.) is attributed to a similar electron configuration around the nitrogen atom. An analogy is drawn between the colour of inorganic salts of these anions (e.g. AgI, HgI_2, $TiBr_4$, ZrI_4, etc.) and the corresponding salts possessing an organic cation.

XII— Diaryltriazen-*N*-sulphonic Acids and their Diazonium Salts
Ibid., 2568–2577

The interaction of diazonium salts with aryl sulphamic acids produces the diaryltriazen-*N*-sulphonic acid salt of the diazonium compound (I)—

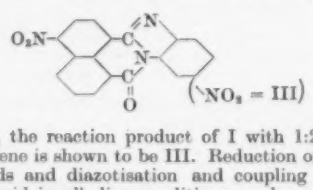


(I)

A comparison of the aryl (Ar′ in I) with the corresponding methyl derivative shows that, in the former case, the fission of the triazen system occurs in neutral, or even in alkaline conditions. An explanation of this fission is given in terms of the electrophilic nature of Ar′. G.J.K.

Direct Azo Dyes— Derivatives of 1:8-Naphthoylen-1′:2′-Benzimidazole
B. M. Krasovitskiĭ, R. M. Matskevich, N. A. Radpchiva, and K. P. Ryazanova
Zhurnal obshcheĭ khimii, **28**, 2485–2489 (Sept. 1958)

The preparation of mono- and dis-azo direct cotton dyes, containing the 1:8-naphthoylen-1′:2′-benzimidazole system, is discussed and the mechanism for the benzimidazole ring formation is suggested. On reaction of 4-nitro-1:8-naphthalene dicarboxylic acid anhydride (I) with *o*-phenylenediamine, II is obtained exclusively—



(II) (NO_2 = III)

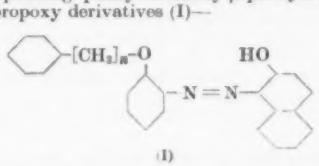
Similarly, the reaction product of I with 1:2-diamino-4-nitrobenzene is shown to be III. Reduction of these nitro compounds and diazotisation and coupling with H or Chicago acid in alkaline conditions produces direct dyes with good fastness on cotton. The disazo dyes possess higher tinctorial value. G.J.K.

Decomposition of Aryldiazonium Borofluorides in Esters of Benzoic Acid
L. G. Makarova and E. A. Gribchenko
Izvestiya Akad. Nauk S.S.S.R., otdel khim. nauk, 693–697 (June 1958)

Phenyl, obtained from the decomposition of benzenediazonium borofluoride, enters into the *meta* position to the carboxyl group when it reacts with the *n*-propyl-, or *n*-butyl ester of benzoic acid; some ester interchange also occurs. In the case of the methyl ester of benzoic acid substitution occurs mainly in the *meta*-, but to a smaller extent in the *para*- position also. The reaction with *iso*-propylbenzoate gives only $C_6H_5COOC_{15}H_{24}$, the structure of which has not been elucidated. In the case of the *p*-bromo- or *p*-nitro-benzene diazonium borofluoride only ester exchange occurs with the benzoic acid ester. The introduction of the aryl grouping from the aryl diazonium borofluoride into the position *meta* to the –COOR group, and also the ester interchange, supports the cationic nature of the attack and the heterolytic character of the decomposition of aryldiazonium borofluoride. G.J.K.

Halogen-substituted Azo Compounds. III— Preparation of *o*-Arylalkoxyanilines
B. I. Stepanov and M. A. Andreeva
Zhurnal obshchei khimii, **28**, 2490–2491 (Sept. 1958)

The azo compound obtained by diazotisation of *o*-chloraniline and coupling with *β*-naphthol is shown, on treatment with benzyl alcohol or its derivatives, to yield the corresponding phenyl-methoxy-*β*-phenylethoxy-, and *γ*-phenylpropoxy derivatives (I)—



(I)

(n = 1, 2, or 3.) Reductive fission yields the corresponding *o*-arylalkoxy aniline. G.J.K.

Constitution of *o*-Hydroxy- and Amino-azo Dyes
A. Schaeffer
Melliand Textilber., **39**, 1002–1005 (Sept. 1958)

Infrared spectra of *o*-hydroxy- and amino-azo dyes, which differ in behaviour from their *p*-isomers and were regarded by Hantzsch *et al.* as *o*-quinone-hydrazones and by Schetty *et al.* as chelates, are interpreted as evidence for the *o*-quinone-hydrazone form, but not the chelate form, except for Naphtol AS derivatives, where the OH chelates not with the azo, but with the carbonyl group. S.M.J.

Oxidative Coppering of *o*-Hydroxyazo Dyes
H. Pfitzner and M. Baumann
Angew. Chem., **70**, 232–8 (1958): *Chem. Abs.*, **52**, 17717 (25 Oct. 1958)

Review of the conversion of *o*-hydroxyazo dyes to the Cu complexes of *o,o′*-dihydroxyazo dyes by oxidation with H_2O_2 in presence of Cu salts at pH 4·5–7·0 and at 40–70°c. The limitations of this reaction, its possible mechanism and its practical significance are discussed. C.O.C.

Spectroscopic Investigation of Two Basic Dyes in Solution
Kh. L. Arvan
Dokladÿ Akad. Nauk S.S.S.R., **121**, 123–125 (1 July 1958)

The spectroscopic investigation of the interaction of Acridine Yellow and Methylene Blue in various solvents has been carried out between 400–700 mμ., particular emphasis being given to the absorption band between 600–700 mμ. It is shown that the spectrum of such a mixture deviates from that expected from a summation of the two individual spectra. Increasing amounts of Acridine Yellow displace the absorption band at about 650 mμ. to longer wavelengths until finally the completely bonded Methylene Blue absorbs at about 685 mμ. From the results obtained, the author concludes that the heat of formation of the complex is 9 kcal./mole (energy of dimerisation of Methylene Blue equals 6 kcal./mole). The competition between aggregates of the individual dyes and complexes is discussed. G.J.K.

Neutral-dyeing Tri- and Penta-cyclic Metallised Azo Dyes
G. Schetty and H. Ackermann
Angew. Chem., **70**, 222–231 (1958): *Chem. Abs.*, **52**, 17718 (25 Oct. 1958)

A general paper, with no experimental details, on polycyclic metal complexes of *o*-hydroxy-*o′*-sulphamoyl and *o*-hydroxy-*o′*-amino azo dyes containing 1 atom of metal per dye molecule (1:1 type). The contribution of the sulphamide H atom to the complex is evident from study of the acid stability, neutralisation curves and absorption spectra of these dyes. They are compared with the usual 2:1 types with respect to their 3-dimensional structures and their wool-dyeing properties. C.O.C.

Reaction between Sodium Tungstate and 4:4′-bis-[(azo-)1-3:4-dihydroxybenzene]-2:2′-stilbenedisulphonic Acid in Aqueous Solutions
K. E. Kleiner and A. Kh. Klibus
Zhurnal obshchei khimii, **28**, 2013–2021 (Aug. 1958)

The reaction between aq. solutions of sodium tungstate and the above bisazo compound has been investigated spectrophotometrically. Between pH 2 and 6 red–brown solutions are obtained containing the azo compound–tungstate complex in the ratio 1:1. In more acid solutions (pH 1) the complex acquires a 1:2 ratio, and blue or blue–violet coloration results ($\lambda_{max} \approx 6{,}000$ A., ε 64,000). The quantitative estimation of tungsten, based on this reaction, is suggested. G.J.K.

Diamine Black BH, C.I. 22590
I. Reichel and E. Berger-Valentin
Acad. rep. populare romine, Filiala Cluj, Studii cercetări ştiinţ., Ser. I, **6**, 199–210 (1955): *Chem. Abs.*, **52**, 17718 (25 Oct. 1958)

In making C.I. 22590 by the asymmetric coupling of tetrazotised benzidine with H acid and 6-amino-2-naphthalenesulphonic acid under slightly alkaline conditions a product is obtained which chromatographic analysis shows to be 75–78% C.I. 22590 and 25–22% Diamine Blue 2B. This mixture has better tinctorial properties than C.I.22590 alone and so it is inadvisable to modify the classical production scheme for this dye in order to obtain a pure product. C.O.C.

Optical Sensitisation of the Photoeffect in Silver Bromide and Thallium Bromide by Organic Dyes
I. A. Akimov
Dokladÿ Akad. Nauk S.S.S.R., **121**, 311–314, (11 July 1958)

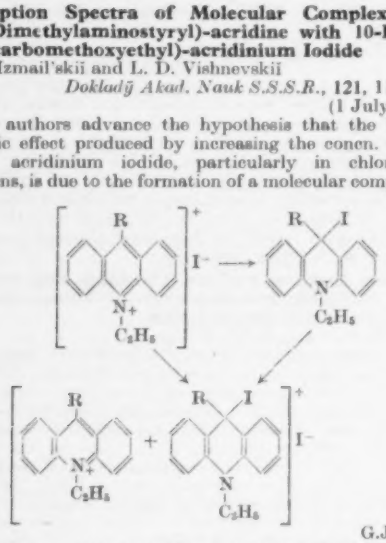
The effect of (*a*) Malachite Green, (*b*) Methylene Blue upon the sensitisation of AgBr and TlBr in the visible and near infrared spectral region is investigated. The results obtained are analogous to those obtained by investigating corresponding iodides (*Dokladÿ*, **102**, 481 (1955)). The author puts forward a mechanism based upon a transfer of the energy of the photon absorbed by the dye to the semiconductor (the bromide), this energy promoting an electron to a localised level in the semiconductor and creating a positive hole; this gives rise to increased conductance. This mechanism demands the presence of an acceptor creating local electronic levels at the surface of the semi-conductor and absorbed Br_2 molecules carry out this function. G.J.K.

Synthesis of Heterocyclic Compounds of Nitrogen. CXI— Photosensitising Dyes. 17
T. Takahashi and K. Sato
Yakugaku Zasshi, **78**, 467–471 (1958): *Chem. Abs.*, **52**, 17257 (25 Oct. 1958)

$C_9H_7N{\cdot}CH_3I$ and 2-$OHCC_5H_4N$ refluxed in presence of ethanol and piperidine yield the greenish purple 1-methyl-2-[2-(pyridyl)vinyl]quinolinium iodide. This in turn when refluxed in ethanol with large excess of CH_3I yields 1-methyl-2-[2-(methyl-2-pyridinium)vinyl]quinolinium diiodide which is also greenish purple. 4-$CH_3C_5H_4N{\cdot}C_2H_5I$ and 3-$CH_3C_5H_4N$ similarly yield the yellow 1-ethyl-4-[2-(3-pyridyl)vinyl]-pyridinium iodide which with large excess of C_2H_5I yields the yellow 1-ethyl-4-[2-(1-methyl-3-pyridinium)vinyl]pyridinium diiodide. 2:3:4-Trimethylthiazolium iodide and 3-$OHCC_5H_4N$ yield the yellow 1:4-dimethyl-2-[2-(3-pyridyl)vinyl]thiazolium iodide which with large excess of C_2H_5I yields the yellow 3:4-dimethyl-2-[2-(3-pyridinium)vinyl]thiazolium diiodide or if refluxed with C_2H_5I yields the yellow 3:4-dimethyl-2-[2-(1-ethyl-3-pyridinium)vinyl]thiazolium diiodide. C.O.C.

Absorption Spectra of Molecular Complexes of 9-(*p*-Dimethylaminostyryl)-acridine with 10-Ethyl-9-(β-carbomethoxyethyl)-acridinium Iodide
V. A. Izmail'skiĭ and L. D. Vishnevskiĭ
Doklady Akad. Nauk S.S.S.R., **121**, 111–114 (1 July 1958)

The authors advance the hypothesis that the bathochromic effect produced by increasing the concn. of the above acridinium iodide, particularly in chloroform solutions, is due to the formation of a molecular complex—



G.J.K.

Fluorescence of Acridine and Acridone Solutions
E. J. Bowen and J. Sahu
J.C.S., 3716–3718 (Oct. 1958)

The fluorescence of acridine (I) in alcoholic soln. is found to be due entirely to the water content of the alcohol, and, while I fluoresces strongly in water, it is non-fluorescent in most org. liquids. The fluorescence intensities diminish with rise of temp. It is found that $(I/F)-K=k\exp(-E/\boldsymbol{R}T)$, where F is the absolute quantum yield of fluorescence, E an "activation energy of fluorescence quenching" (cal./mole), T the absolute temp., and K and k are constants. It is concluded that a hydroxylic environment tends to hold the excited acridine mol. in a rigid form where it has difficulty in losing energy by non-radiational processes. Soln. of acridone in org. solvents are more fluorescent than those of I; in methyl, ethyl, and *iso*propyl alcohols, and in water, over the temp. range −70° to +20°, the quantum yields of fluorescence are practically unity. H.H.H.

Tetra-2-benziminazolylethylene, a new Yellow Chromophore
R. G. Arnold, R. S. Barrows, and R. A. Brooks
J. Org. Chem., **23**, 565–568 (1958)

The strong colour and stability of tetra-2-benziminazolylethylene and its derivatives make them potentially useful as dyes and pigments. C.O.C.

Thiacarbocyanines with Unsaturated Groups as Substituents
M. A. Al'perovich, I. K. Ushenko, and L. N. Tyurina
Zhurnal obshcheĭ khimii, **28**, 2538–2547 (Sept. 1958)

The reaction of aryl acrylic acids or coumarine with diazotised 2-methyl-5-amino-, and 2-methyl-6-aminobenzthiazoles produces 7 new benzthiazole derivatives with unsaturated groups in the 5- or 6-position; the latter on reaction with alkylating agents give the corresponding quaternary ammonium salts (I). Condensation of I with ortho esters of carbonic acids in pyridine yields 14 new thiacarbocyanines with unsaturated groups in the 5- or 6-position. The presence of such unsaturated groups is shown to cause a bathochromic displacement of the absorption band. G.J.K.

Direct Oxidation of Indoles to Oxindoles
C. E. Dalgliesh and W. Kelly
J.C.S., 3726–3727 (Oct. 1958)

Under the usual strongly alkaline conditions, skatole is converted by persulphate into a large number of products which indicate the opening of the pyrrole ring, but, under mildly acid conditions (pH 4–5), the major product (38%) is 3-methyloxindole. 3:5-Dimethylindole similarly oxidises to 3:5-dimethyloxindole, but indole oxidises at the 3-position to give indican and not oxindole. H.H.H.

2-Methyl-4-alkoxychromone Cyanine Dyes
A. Szuchnik, J. Świderski, and W. Czerska
Roczniki Chem., **32**, 255–6 (1958): *Chem. Abs.*, **52**, 17718 (25 Oct. 1958)

Phenylacetate (170 g.) is dropped in 1 hr. into anhydrous $AlCl_3$ (153) and then heated for 3 hr. at 180°c. Dil. HCl (500 ml.) is then added, the mixture steam distilled and the distillate extracted with ether. The fraction of the extract, b_{10} at 92·4°c. or b_{15} at 96–98°c., is collected. There is a 35–40% yield of *o*-hydroxyacetophenone (I). The residue after I has been steam distilled off is *p*-hydroxyacetophenone (II), which is dissolved in saturated aq. Na_2CO_3 (0·5 mole/mole of II), filtered, boiled with charcoal and saturated with CO_2. The precipitated II is recrystallised to give a 15–20% yield. A mixture of dry I (40 g.) and ethyl acetate (78) is dropped into Na metal (20) at the b.p. of the mixture. Ice (500) and small excess of acetic acid are then added. *o*-Hydroxyacetophenone is extracted with ether, excess ethyl acetate is distilled off, acetic acid (140 ml.), acetic anhydride (10), and conc. HCl (10) are added and the mixture heated to 100°c. for 30 min., boiled, diluted with water and neutralised with $NaHCO_3$. The resulting 2-methyl-4-chromone (IV) is crystallised as the hydrochloride, decomposed with $NaHCO_3$, and recrystallised from gasoline to give a 50% yield. 4-Methoxy-2′-methyl-2:4′-chromone cyanine (V) iodide is prepared by heating IV (8 g.) with dimethylsulphate (6·5) at 70–80°c., KI (16·6) in water (25 ml.), and several crystals of $Na_2S_2O_3$ are then added and the V is filtered off and recrystallised from methanol. V perchlorate is prepared from V sulphate by reaction with saturated aqueous $NaClO_4$ and crystallisation from ethanol. 4-Ethoxy-2′-methyl-2:4′-chromone cyanine iodide (VI) and perchlorate (VII) are similarly prepared using diethyl sulphate. VI is recrystallised from a 1 : 1 mixture of ethanol and ethyl acetate, VII is recrystallised from ethanol. C.O.C.

Dipole Moments of Hemioxanin Derivatives of 3-Ethylrhodanine and 1:3-Indandione
E. A. Shott-L'vova, Ya. K. Syrkin, I. I. Levkoev, and M. V. Deichmeister
Doklady Akad. Nauk S.S.S.R., **121**, 1048–1051 (21 Aug. 1958)

The authors show that the change in the position of the max. absorption band of these dyes in ethanolic solution on elongation of the polymethine chain can be related to the change in μ. A series of 17 polymethine dyes and the corresponding μ. and λ_{max} values are tabulated. It is shown that the presence of an acetyl-phenyl-amino group minimises the change both of λ_{max} and of μ. on chain elongation; on the other hand, the 1:3-indandione derivatives exhibit the greatest bathochromic displacement and change of μ. The presence or absence of internal hydrogen bond is discussed. G.J.K.

Anthraquinone and Anthrone Series. XXII— 3:4:9:10-Dibenzopyrene and Pentaphene from Indanthren Scarlet 4G (C.I. 59115) and Mayvat Brilliant Red AF (OBM)
F. B. D. Tilak, M. K. Unni, and K. Venkataraman
Tetrahedron, **3**, 62–7 (1958): *Chem. Abs.*, **52**, 18345 (10 Nov. 1958)

Mayvat Brilliant Red AF is shown to be a halogenated derivative of 3:4:9:10-dibenzopyrene-5:8-quinone obtained by dehalogenation of Mayvat Brilliant Red AF or Indanthren Scarlet 4G (C.I. 59115) by reduction with Raney Ni in aqueous NaOH. The differences in hue of these two dyes and the different colours they give in H_2SO_4 are caused by the number and positions of the halogen atoms in them. C.O.C.

Synthesis of Carboxylic Acid Hydrazides and *s*-Triazoles of the Anthraquinone Series
E. Klingsberg
J. Amer. Chem. Soc., **80**, 5786–9 (5 Nov. 1958)

An account of the synthesis of 2-anthraquinonecarboxylic acid hydrazide, its 1-chloro and 1-amino derivatives and certain related diaroylhydrazines, and of the conversion of some of these compounds to *s*-triazoles. All these compounds are of interest because of their relationship to anthraquinonyl oxadiazoles which have recently acquired importance as vat dyes (see *USP* 2,464,831, 2,749,352, and 2,759,948). C.O.C.

Chemistry of Polynuclear Compounds

I— Infrared Spectra of some Polynuclear Quinones and Polynuclear Aromatic Hydrocarbons

R. A. Durie, R. E. Lack, and J. S. Shannon

Australian J. Chem., **10**, (4), 429–447 (1957)

Polynuclear quinones are adopted as models for an investigation into the coking mechanism of coal, and their infrared absorption spectra (between 2000 and 650 cm^{-1}) are now reported, namely for: dibenzanthrone (I); *iso*-dibenzanthrone (II); 16:17-dimethoxydibenzanthrone (III); 16:17-diethoxydibenzanthrone (IV); pyranthrone (V); flavanthrone (VI); 4:10-dibromoanthanthrone (VII); 4:4′-dibenzanthronyl (VIII); 2:2′-dimethyl-1:1′-dianthraquinoyl (IX); and for the polynuclear aromatic hydrocarbons violanthrene (X) and *iso*violanthrene (XI). Factors influencing the quinonoid carbonyl group bond order (hence frequency of absorption) and the redox potential are discussed. Tentative correlations are advanced for strong absorption bands in the CH out-of-plane vibration region. The mol. non-planarity of VIII and IX are indicated.

II— Behaviour in Concentrated Sulphuric Acid Solution and Absorption Spectra of some Polynuclear Quinones

R. A. Durie and J. S. Shannon

Ibid., **11** (2), 168–188 (1958)

The electronic absorption spectra of dibenzanthrone (I), and 16:17-dimethoxy- (II), 16:17-diethoxy- (III), 16-methoxy-17-ethoxy (IV), 16-methoxy-17-hydroxy- (V), and 16-ethoxy-17-hydroxy- (VI) dibenzanthrone in chlorobenzene and dioxane soln., and for I, II, V, and VI in conc. H_2SO_4, are reported and discussed. Non-planar configurations are proposed for II–VI, and the possibility of the same for I considered. IV, V, and VI are new vat dyes; V is produced by the action of conc. H_2SO_4 on II and IV, and VI from III. IV is prepared by the methylation of VI. For comparison purposes, the corresponding spectra in conc. H_2SO_4 are reported for anthraquinone, and its 1-amino-, 2-amino-, 1:2-diamino, and the 1:4-, 1:5-, and 1:8-dimethoxy- deriv. The infrared spectra for the new dyes IV, V, and VI confirm the identification of these cpd. as 16:17- disubst. dibenzanthrones and establish the nature of the alkoxyl substituents. The quinonoid-carbonyl frequencies are discussed.

III— Structures and Absorption Spectra of 1:1′-Dianthrimide and Indanthrone

R. A. Durie and J. S. Shannon

Ibid., 189–199 (1958)

The vibrational and electronic absorption spectra of indanthrone (I), 1:1′-dianthrimide (II), and 1:2:2′:1′-anthraquinonecazine (III) are presented and discussed. The absorption spectra of I and II, together with other cited evidence, favour the tetraketo form for I rather than the enol form recently proposed by Wyman (*J. Amer. Chem. Soc.*, **78**, 4599–4604 (1956)). H.H.H.

Phthalogens (FBy) and Phthalocyanines

J. Eibl

Melliand Textilber., **39**, 522–527 (May), 660–663 (June), 772–775 (July 1958)

An extensive review covering much of the same ground as Gund (J.S.D.C., **69**, 671–681 (Dec. 1953)), but giving considerably more detail on discovery, formulae of all intermediates and dyes, and reactions such as hydrolysis, alcoholysis, salt formation, reaction with aldehydes, transamination, and aminolysis. In addition, dyeing, as distinct from printing, of Phthalogen Brilliant Blue IF3G, and heavy-metal poly*iso*indolenines such as Phthalogen Brilliant Blue IF2GK are dealt with. S.M.J.

Photochemical Stability of Phaeophytin-*a* and -*b* and of their Metal Derivatives

I. L. Kukhtevich

Ukrain. khim. zhurnal, **24**, 336–339 (1958)

The photochemical stability of phaeophytin-*a* and -*b* and of their Cu, Zn, Co, Cd, Ag, Hg, and Pb derivatives has been studied, and phaeophytin-*a* and its derivatives are shown to be less stable. The stability of the metal derivatives is shown to decrease, within both series, in the order: Cu > Co > Ag > Pb > Hg > Zn > Cd; the latter two derivatives being very readily affected photochemically. Fading in the blue-violet and red regions of the spectrum occurs more readily in the metal derivatives. G.J.K.

Cyanine Dyes from 2-Methylacenaphthene-thiazole

A. V. Stetsenko and A. D. Kurbatova

Ukrain. khim. zhurnal, **24**, 354–357 (1958)

The synthesis of 2-methylacenaphthene-5′:4′,4:5-thiazole and its quaternary ammonium iodide (I) has been carried out. The reaction of I with various aromatic or heterocyclic tertiary bases yields 8 new cyanine dyes; the absorption maxima of which are listed. G.J.K.

New Intermediates and Dyes. VI— The Chemistry of 2-*n*-Butylanthraquinone. Derived Dyes for Synthetic Fibres

A. T. Peters, Jun., and A. T. Peters

J.C.S., 3497–3503 (Oct. 1958)

Many deriv. of 2-*n*-butylanthraquinone (I) are described and, in particular, the nitro group in its 1-nitro- deriv. has been replaced to give a series of alkylamino- and arylamino- deriv., which are dyes for cellulose acetate rayon, nylon, and Terylene; apart from a slight bathochromic effect, especially on nylon, the shades and fastness of the dyeings were only slightly affected by the butyl group. Dinitration of I is described in detail, and the derived 1:5- and 1:8-diamino- and -bismethylamino-quinones are compared with the bluer 1:4-disubst. analogues, prepared from 1-amino- and 1-methylamino-4-bromo-2-*n*-butylanthraquinones, respectively. On cellulose acetate rayon, dyeings of the three diamino-2-*n*-butylanthraquinones showed the deep reddish violet of the 1:4-diamine, the 1:5- and 1:8-diamines affording reddish-orange and orange-red shades respectively. Replacement of the amino- by the methylamino- group afforded a bathochromic effect in all cases, the 1:4-, 1:5-, and 1:8-deriv. giving blue, bluish-red, and reddish-violet shades respectively. H.H.H.

Some Properties of the Ground Triplet State of Chlorophyll (C.I. Natural Green 3) and Related Compounds

R. Livingston and E. Fujimori

J. Amer. Chem. Soc., **80**, 5610–3 (5 Nov. 1958)

The absorption spectra of the lowest triplet states of certain polyatomic molecules were measured by use of the flash photolytic-flash photographic technique. The compounds investigated included chlorophylls-*a* and -*b*, pheophytins-*a* and -*b*, Zn-chlorophyll-*a*, several porphyrins and Mg-phthalocyanine. In general their spectra resemble those already published for the triplet state of the chlorophylls. The principal absorption maxima of the ground state do not appear in the absorption of the triplet state where they are replaced by a broad absorption, having a maximum on the long wavelength side of the Soret band and gradually decreasing toward the red. No transients were detectable for Cu or Fe substituted porphyrins or for Cu-chlorophyll-*a*, presumably because of the short lives of the triplet state of those molecules. C.O.C.

Spectrophotometric Study of the Reaction between Chlorophyll and Ferric Chloride

S. S. Butsko and B. Ya. Dain

Zhurnal obshchei khimii, **28**, 2603–2611 (Sept. 1958)

The reaction of chlorophyll with ferric chloride in ether and acetone solutions results in the displacement of Mg and the formation of ferric phaeophytin. The reaction occurs extremely slowly and is characterised by a high temp. coefficient. In acetone solution, this reaction leads to the formation of an intermediate (grey) product the spectrum of which is typical of a dihydroporphin derivative. The change of the intermediate product to ferric phaeophytin occurs irreversibly. Ferrous ions strongly catalyse the penetration of iron into the chlorophyll nucleus, whilst oxygen has a retarding effect. A mechanism based on the presence of $[Fe(MgPh)]^{3+}$ ions is put forward. G.J.K.

Titanium Dioxide Production in the U.S.A. and its Consumption by Various Industries

Anon.

Chem. Eng. News, 30 (24 Nov. 1958)

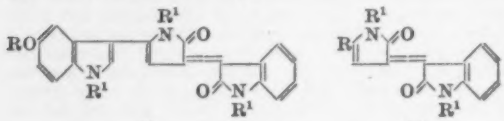
Production of TiO_2 in the U.S.A. in 1957 was about 400,000 short tons. The following was the consumption in thousands of short tons by various industries: paint, varnish, and lacquer 230·8, paper 56·8, floor covering 20·0, coated textiles 16·4, rubber 18·4, printing ink 7·6, other 50·1. C.O.C.

Synthesis of Violacein and Related Compounds

J. A. Ballantine, R. J. S. Beer, D. J. Crutchley, G. M. Dodd, and D. R. Palmer

Proc. Chem. Soc., 232–233 (Aug. 1958)

Using methods which are outlined, the indolyloxindolylidene-2-oxopyrroline (II; R = 3-indolyl, R^1 = H), which resembles violacein (I; R = R^1 = H) in many important respects, and also the 5-methoxyindole analogue (I; R = CH_3, R^1 = H), have been prepared. Methylation of the latter cpd. affords tetramethylviolacein, identical with a sample derived from the natural pigment. Demethylation of the 5-methoxyindolyloxopyrroline with HBr in acetic acid gives violacein. A recent pigment isolated from *Chromobacterium violaceum* is identified as deoxyviolacein (II; R = 3-indolyl, R^1 = H).



(I) (II)

H.H.H.

Pigments of Low Refractive Index

S. Lavren and C. R. V. Linden

Paint Varnish Production, **48** (8), 39–41, 70 (1958)

Synthetic hydrated Ca silicates are obtained by a hydrothermal reaction of diatomaceous silica, hydrated lime and water under high pressure. Properties and possible uses in paint are listed for the various products, the most promising fields seeming to be in poly(vinyl acetate) emulsion paints and paints for exterior decorating. C.O.C.

Some Passivating Properties of Pigments

I. L. Rosenfel'd, F. I. Rubinshteĭn, and V. V. Zhebrovskiĭ

Izvestiya Akad. Nauk S.S.S.R., otdel. khim. nauk, 679–683 (June 1958)

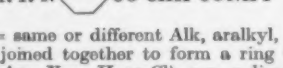
The passivating properties of chromate pigments have been studied. The irreversible electrode potential of steel in thin layers of aq. extracts of pigments is displaced by 200–300 mv. to the positive side, becoming stable with time. This potential greatly depends upon pigment nature, and the following series, in decreasing order of passivating ability, has been established: mixed Ba-K-chromates (technical) > mixed Ba-K-chromates (chem. pure) > Sr-chromate > Zn-chromate. It is shown that the difference in the passivating properties of the pigments is due to different solubility of the passivating portion of the pigments. Concn. of CrO_3 in the extract of the mixed chromate is considerably higher than in that of Sr and Zn-chromate (10–13 g. per litre instead of 0·5 g. per litre). G.J.K.

PATENTS

Substituted Benzoylacetanilides as Colour Couplers

ICI *BP* 805,505

Colour couplers having very low absorption of green light and marked absorption of ultraviolet radiation so that unreacted coupler remaining after colour development serves to absorb ultraviolet radiation and so protect the yellow azomethin dye image from fading are of formula—



(R^1 and R^2 = same or different Alk, aralkyl, Ar or *cyclo*alkyl or are joined together to form a ring with the N atom; Y = Ar; X = H or Cl), e.g. *p*-dimethylaminobenzoylacet - (2 - *N* - methyl - *N* - octadecylamino - 5 - carboxy)anilide. C.O.C.

Monoazo Pigments from Arylides of 3-Hydroxy-2-Naphthoic Acid

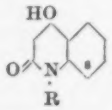
FBy *BP* 804,673

The monoazo pigments obtained by coupling diazotised 4-acetamido-2-nitroaniline (which may be further substituted in the benzene nucleus by Hal, Alk, or OAlk) with arylides of 3-hydroxy-2-naphthoic acid are oranges, maroons, red-browns, etc. of good fastness to light and solvents; they may also be produced on textiles by azoic processes. Thus diazotised 4-acetamido-2-nitroaniline is coupled with a suspension in dilute aq. acid of 3-hydroxy-2-naphtho-*o*-anisidide to give a maroon. E.S.

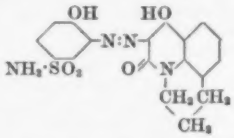
Red and Brown Metal(Chromium and Cobalt)-complex Monoazo Wool Dyes having a Quinoline Nucleus

FBy *BP* 804,766

Sulphonamides or alkylsulphonyl derivatives of *o*-aminophenol are diazotised and coupled with quinoline derivatives



(R = Alk, phenyl, or benzyl, or is $-CH_2{\cdot}CH_2{\cdot}CH_2-$ linked to the adjacent 8-position) to give monoazo compounds whose Cr and Co complexes are red and brown wool dyes applicable from a neutral or weakly acid bath. Thus 1:8-trimethylene-4-hydroxycarbostyril, obtained by heating tetrahydroquinoline with malonic ester, coupled with diazotised 2-aminophenol-4-sulphonamide gives

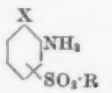


Stirring with aq. ammoniacal $CoSO_4$ at 70–80°c. gives the Co complex which dyes wool orange-brown. E.S.

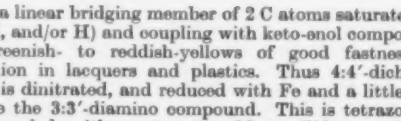
Metal-complex Monoazo Pigments for Spin-dyeing Thermoplastic Fibres

S *BP* 805,217

The Co and Cr complexes of monoazo compounds obtained by diazotising amines—



(R = phenyl or an amino group in which one or both H atoms may be substituted by Alk, cycloalkyl, aralkyl or Aryl, and the 2 Alk groups may be cyclically combined to give e.g. a piperidine or morpholine group; X = OH or OCH_3), and coupling with 3:2-hydroxynaphthoic acid or its chloro-, bromo-, methyl-, or methoxy- derivatives, are bordeaux, violets and blues highly soluble in polar solvents, e.g. acetone, and are very suitable for inclusion in the spinning solutions of cellulose acetate and polyester and polyacrylonitrile fibres. Thus 2-aminophenol-4-sulphon-*γ*-methoxypropylamide is diazotised and coupled with a soln. in aq. NaOH of 3:2-hydroxynaphthoic acid to give



Heating at 100°c. with chrome alum in formamide gives the Cr complex, which is a violet. E.S.

Yellow Disazo Pigments

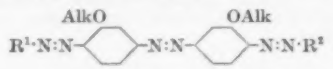
Gy *BP* 803,985

The disazo pigments obtained by tetrazotising diamines

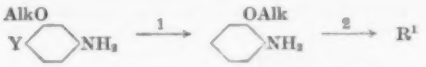
(X = a linear bridging member of 2 C atoms saturated by O, OH, and/or H) and coupling with keto-enol compounds are greenish- to reddish-yellows of good fastness to migration in lacquers and plastics. Thus 4:4′-dichlorobenzil is dinitrated, and reduced with Fe and a little HCl to give the 3:3′-diamino compound. This is tetrazotised and coupled with acetoacet-*p*-chloroanilide to give a greenish yellow. E.S.

Blue and Grey Metal(Copper)-complex Trisazo Direct Dyes
FBy *BP* 803,810

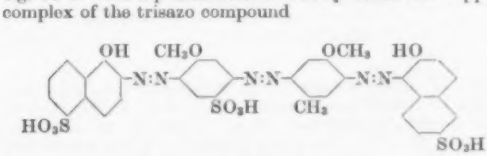
The Cu complexes of trisazo dyes—



(R^1, R^2 = residues of *o*-coupling naphtholsulphonic acid which may have an acylamino group as substituent; the benzene rings may contain Alk, OAlk, Hal or SO_3H; 3 water-solubilising groups such as SO_3H or COOH are present) dye cellulose blue or grey. Two methods of making the metal-free dyes are available: (1) A 3-alkoxyaniline with a substituent Y in the 4-position capable of conversion to NH_2 (e.g. NO_2 or NHAcyl) is diazotised and coupled with an *o*-alkoxyaniline, and the disazo dye so formed is then diazotised and coupled with a naphtholsulphonic acid—



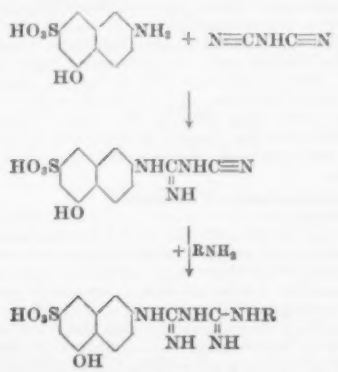
Y is converted into NH_2 which is then diazotised and coupled with R^3. (2) A 2-alkoxyaniline with Y in the 4-position is diazotised and coupled with R^1, Y is then converted into NH_2, which is diazotised and coupled with an *o*-alkoxyaniline, the aminodisazo dye is diazotised and coupled with R^2. Coppering is carried out by heating at e.g. 90°c. with aq. ammoniacal $CuSO_4$. Thus the copper complex of the trisazo compound



dyes cotton and viscose greyish blue. E.S.

Biguanide Derivatives of Naphthol Sulphonic Acids
American Cyanamid Co. *USP* 2,826,606

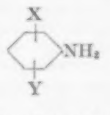
Dye intermediates are prepared by (1) reacting dicyanamide with J-acid to give a substituted cyanoguanidine and (2) reacting this product with a second mol. J-acid or a different aminonaphthol.



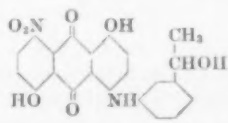
(R = hydroxynaphthyl residue). Thus, a 40·2% paste of J-acid (243·9), Na dicyanamide (17·8), and water (200) are refluxed for 22 hr. (pH = 4·6). The solution is then made slightly alkaline with Na_2CO_3 and salted with NaCl (120). The product is separated by cooling, filtering, and washing with (*a*) salt solution, and (*b*) alcohol. A brilliant red direct dye is then obtained as follows. A solution of diazotised 4-aminoazobenzene-3:4′-disulphonic acid (8·6) in water (100) is added to the bis-J-acid-biguanide (5·86) in water (200). During coupling, $NaHCO_3$ (8·4) in water (150) is added and stirring continues overnight. NaOH (35) and NaCl (240) are added and the product is filtered and washed with alk. salt solution. E.T.

Anthraquinone Disperse Dyes
Celanese Corpn. of America *USP* 2,827,356

Blue cellulose acetate dyes, fast to gas fumes and light and yielding dyebaths which are stable over a wide pH range, are derived by condensing hydroxy-polynitroanthraquinones (with $\leq$ 1 NO_2 *para* to an OH group) with aromatic primary amines so that the product still contains a NO_2 group. Reaction occurs, preferably between 110°c. and 145°c. during 6–18 hr. and it is advantageous to use a solvent boiling between these temperatures so that strict temperature control can be achieved, without difficulty, by refluxing. Especially valuable products are obtained by using an aromatic amine of formula—



(X = $(C_nH_{2n})OR$ or $O-(C_nH_{2n})OR$ (*n* = a small integer; R = H, Alk, hydroxyalk, hydroxyalkoxy); Y = H, Alk, alkoxy, OH, Hal, COOH, carbethoxy, CN, NH_2, alkylamino, hydroxyalk, hydroxyalkoxyalk). Thus, 4:8-dinitroanthrarufin (33) in ethyleneglycol monomethylether (480) is reacted with *m*-aminophenylmethylcarbinol (68) by refluxing 18 hr. The cooled melt is poured into 1·5% HCl (1500) and filtration and water-washing until the filtrate is colourless follows. Purification is effected by dissolving the crude product in ethyl alcohol (1600), re-precipitating by pouring the solution into 20% HCl (1000), filtering and washing. The cake is then slurried with 2·5% Na_2CO_3 solution (2000), re-filtered and washed until any brown material present is removed. The product is



Of especial interest, because of their high affinity, are mixtures containing $\leq$ 25% of 1:5-dihydroxy-4-(methylcarbinol)-anilino-8-nitro- and 1:8-dihydroxy-4-(methylcarbinol)-anilino-5-nitro-anthraquinones. E.T.

Anthraquinone Dye Developers
International Polaroid Corpn. *BP* 804,973

Dyes of formula —

$Y_{(3-m)}\cdot A\cdot(NQ\cdot Z\cdot D)_m$ or $Y_{(3-m)}\cdot A\cdot(NQ\cdot D)_m$

(A = an anthraquinone nucleus; D = Ar containing at least two hydroxyl, amino or subst. amino groups so that the dye can develop exposed silver halide; Q = H or Alk or, when the substituents on the Ar of D are hydroxyls, aliphatic or aromatic acyl; Z = bivalent organic radical containing at least one CH_2 group; *m* = 1–5; Y = H, Hal, amino Alk, Ar, nitro, alkylamino, arylamino, aryloxy, alkoxy, hydroxyl, sulphonamido, carboxamido, carboxy, sulpho, –NQ·D or –NQ·Z·D), in addition to imparting an image-receiving element a dye image also develops a latent silver halide image in an exposed photographic layer. Thus 1:4-bis-(2′:5′-dihydroxyanilino)-anthraquinone used in a transfer process gives a greenish cyan positive dye image. Similarly *N*-mono-benzoyl-1:4-bis[β-(3′:4′:dihydroxyphenyl)-ethylamino]-anthraquinone gives a magenta positive dye image. C.O.C.

Black Vat Dyes requiring no Aftertreatment
Southern Dyestuff Corpn. *USP* 2,831,871

Vat dyes giving blacks on cotton without any aftertreatment are made by reacting compounds of the dibenzanthrone series in 75–100% H_2SO_4 at 0–75°c. with Na, K or NH_4 nitrate. Thus, technically pure dibenzanthrone (46) is dissolved in 98% H_2SO_4 (500); $NaNO_3$ (10) is added and the mixture is stirred at 0–3°c. for 4 hr. The mixture is drowned into water (5000) and the product separated by filtration and washing. Halogeno- and dihalogeno-derivatives of benzanthrone or *iso*dibenzanthrone may also be used, and other suitable nitrates are the hydrated nitrates of Hg, Cu, Mg, Ce, Al, Bi; also used are the nitrates of Li, amyl, urea, and Ca. E.T.

Dye Developers

International Polaroid Corpn. *BP* 804,974

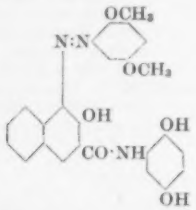
Compounds of formula $D^1{\cdot}X{\cdot}D^2$ (D^1 = the chromophoric system of a dye; D^2 = a group containing an Ar nucleus substituted by > 1 hydroxyl, amino or substituted amino groups; X = an achromophoric bond which does not contribute a colour-producing group to the molecule but acts to prevent or interrupt any system of conjugation or resonance extending from D^1 to D^2) are dye developers whose colour characteristics are the same as those of the dye from which they are prepared, thus *p*-aminoazobenzene (C.I. 11000) and *o*-monobenzoyl-hydroquinonylamino-dichloro-*s*-triazine are dissolved in anhydrous acetone and treated for some hours with aqueous Na_2CO_3. The cyanurated reaction product is hydrolysed by heating, under N_2 in 50% aqueous methanol containing 5% NaOH for 5 min. and then acidifying. The resulting 2-hydroquinonylamino-4-(*p*-phenylazoanilino)-6-hydroxy-*s*-triazine gives a yellow positive dye image when used in a transfer process.

BP 804,975

Compounds of formula—

$$R^1{\cdot}N{:}N{\cdot}R^2{\cdot}CO{\cdot}NH{\cdot}(Alk\ of\ 1\text{–}5\ C)_n{\cdot}D^2$$

(R^1 = subst. or unsubst. Ar; R^2 = Ar substituted by OH *ortho* to the carboxamido group; n = 0 or 1), e.g.

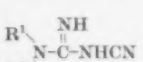


obtained by coupling 2:5-dimethoxyaniline with 2-hydroxy-*N*-(2′:5′-dihydroxyphenyl)-3-naphthamide, are used as dye developers. C.O.C.

Stable Compositions of Sulphuric Acid Half Esters of *leuco*-Vat Dyes

American Cyanamid Co. *USP* 2,827,358

Earlier attempts to stabilise sulphuric acid half esters of *leuco*-vat dyes, which are subject to decomposition by acids, were by adding (*a*) moderate amounts of alkali, e.g. Na_2CO_3; (*b*) excess alkali; and (*c*) urea. Method (*a*) failed to impart stability; method (*b*) led to weak and spotty dyeings and prints by retarding dye development during the HNO_2, chromic acid etc. treatment stage, and method (*c*) gave rise to dye preparations which caked on standing. Effective stabilisation without the above disadvantages is now achieved by using a small amount of alkali, an agglutinant, e.g. dextrine, carbohydrates, glue, etc., and 1–15% on the weight of the vat dye of a dicyandiamide of formula—



(R^1, R^2 = H, Alk, hydroxyalk, aralkyl, aryl). Thus, an alkali metal salt of the *leuco* sulphuric acid half ester of a vat dye in filter press cake form (400 pt. containing 100 pt. dye solids) is intimately mixed with dextrine (20), methylol dicyandiamide (15) and NaOH (5). The paste is dried under vacuum at 60°c. and, on testing, shows no decomposition or caking after 100 days' heating in a closed bottle at 65°c. E.T.

Metal-free α- and γ-Phthalocyanines Stable to Solvents

BASF *BP* 805,514

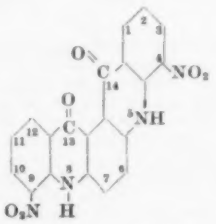
The unstable forms of α- and γ-metal-free phthalocyanines are ground with inorganic substances stable to chlorine, the mixture chlorinated in a fluidised bed at 0–100°c. to a chlorine content of 1–10%, if desired the chlorinated product mixed with sufficient of the corresponding solvent-unstable phthalocyanine to bring the total chlorine content to 1–3%, and finally the resultant product is brought into a state of fine division. C.O.C.

4:9-Dinitroquin-(3:2-a)-Acridine-13:14(5:8)-Dione Pigment

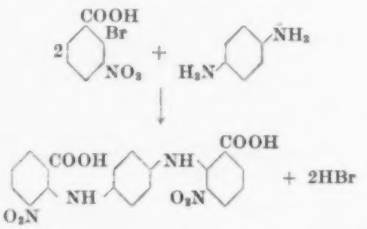
DuP *BP* 805,247

USP 2,830,990

A yellow-to-orange pigment having excellent light fastness and substantial resistance to bleeding in solvents is of formula—



The intermediate for its production, viz. *N*:*N*′-di-(2-carboxy-6-nitrophenyl)-*p*-phenylenediamine, is made by the reaction



Thus, to cupric acetate (4) in ethyleneglycol (1350) are added 2-bromo-3-nitrobenzoic acid (196·8), K_2CO_3 (110·4) and *p*-phenylenediamine (43·2). Heating at 35–45°c. (1·5 hr.), 60–80°c. (2 hr.) and 125–135°c. (3 hr.) is followed by dilution with water (1200) and filtration to remove impurities. The product (78) is separated by adding glacial acetic acid (80), filtering, washing acid-free and drying. At this stage only 1 mol. of each starting material has reacted and the above product (105·3), cupric acetate (1) in ethylene glycol (900), more 2-bromo-3-nitrobenzoic acid (95) and K_2CO_3 (86) are heated in 3 hr. to 140°c. and held at 140°c. for 3 hr. Dilution, filtration and separation follow as before to give the intermediate (116) of m.p. 280°c. Formation of the pigment then follows by ring closure. Thus, the intermediate (52) and $POCl_3$ (260) are refluxed (1·5 hr.), most of the $POCl_3$ is removed by vacuum distillation and the remaining melt is added slowly to water (1500) and stirred 1·5 hr. The solid is filtered, washed acid-free and reslurried at 40°c. for 1 hr. with water (3000) and Na_2CO_3 (30). After filtration and washing alkali-free the product is dried (38) and then acid pasted to give the final pigment having surface area > 60 sq.m./g. E.T.

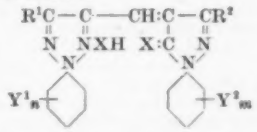
Metal Phthalocyanine Pigments

B. F. Goodrich Co. *USP* 2,824,108

Specially prepared metallic oxides for use as catalysts in the formation of metal phthalocyanines are claimed as giving higher yields than those obtained with the usual form of metallic oxide catalyst. Especially useful are hydrated oxide gels of Ti and Zr. Thus, e.g. $TiCl_4$ is added dropwise to 40 × its volume of water and is hydrolysed to give an acidic translucent sol; addition of NH_4OH to pH 9–10 gives a white gel— $TiO_2{\cdot}XH_2O$ or $Ti(OH)_xO_{2-x/2}$ (x = 1–4). The slurry is filtered through muslin to recover the gel which is then water-washed free from Cl ions by decantation. (To this wash-water is added HNO_3 (0·8 ml. of 6 *N* acid/ml. $TiCl_4$) and NH_4OH (1 ml. 6 *N*/ml.) to maintain the gel structure.) Final distilled water washing removes any mechanically-held solution of NH_4NO_3. Such a freshly prepared gel contained 4·7% TiO_2/95·3% water. Phthalocyanine formation followed by heating trichlorobenzene (4·5 mol.), tetrachlorophthalic anhydride (0·2 mol.), urea (1·33 mol.), cupric ion from 13·5 g. $Cu(NO_3)_2{\cdot}3H_2O$ (0·056 mol.), and TiO_2 from 3·7 g. of the gel (0·0466 mol.) at 175–85°c. Separation of the product by filtration and washing (details given) resulted in a yield of 89·2% of theory. E.T.

Methylidyne bisPyrazolones as Yellow Disperse Dyes
General Aniline *USP* 2,840,443

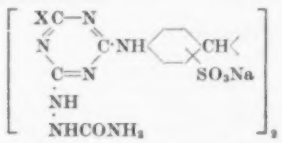
Compounds of formula—



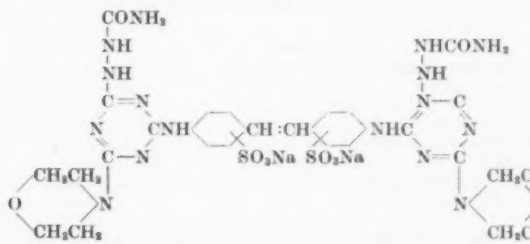
(R^1 and R^2 = aliphatic or monocyclic aromatic radicals; X = O or S; Y^1 and Y^2 = H, Hal, alkoxy, Alk or sulpho amido; n and m = 1 or 2) are disperse dyes yielding fast yellows on wool, silk, acetate rayon and synthetic fibres such as nylon. Thus 4:4′-methylidyne bis(1-phenyl-3-methyl-5-pyrazolone) yields greenish yellow dyeings of excellent fastness to washing and light on Dacron and excellent fastness to washing and good fastness to light on Dynel. C.O.C.

Triazinostilbene Fluorescent Brightening Agent
General Aniline *USP* 2,840,557

Compounds of formula—



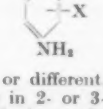
(X = morpholino, dimethylamino, diethylamino- or di-(hydroxyethyl)-amino), e.g.



are fluorescent brightening agents, having bluish fluorescence over a wide pH range, substantive to cellulose, stable to hypochlorite and applicable from soap or detergent baths. C.O.C.

Polyamine Oxidation Dyes for Animal Fibres
Société Monsavon-L'Oréal *BP* 805,746

Polyamines of formula—



(R^1 and R^2 = same or different, H or β-hydroxyethyl; X = primary amino in 2- or 3-position), e.g. 1:2:4-triaminobenzene, 1-(β-hydroxyethyl)-amino-2:4-diaminobenzene, 1-(β-hydroxyethyl)-amino-3:4-diaminobenzene and 1-di-(β-hydroxyethyl)-amino-3:4-diaminobenzene, when used as oxidation dyes, e.g. for human hair, develop rapidly in air without need to use hydrogen peroxide or other oxidising agent. Thus application of an aqueous solution of 1:2:4-triaminobenzene at pH 8 quickly dyes the hair bluish black, if the solution is at pH 6–7 then a slightly reddish black results. C.O.C.

***cyclo*Diene-Halogenosilane Treated Mineral Pigments**
Esso Research & Engineering Co. *BP* 805,512

Treatment of hydrated silica clays, calcium silicate or alumina with a halogenosilane of formula $R_mSi_nX_{2n+2-m}$ (R = *cyclo*alkadienyl radical; m = 1 to $2n+1$; n = 1–5; X = Cl, Br or I) are readily compounded with saturated resins, e.g. polyethylene and poly(vinyl chloride), though they are not able to form bonded pigment-resin particles, or with rubbers, plastics, paints, and inks containing such resins. C.O.C.

Metal Phthalocyanine Pigments
B. F. Goodrich Co. *USP* 2,825,733

Metal phthalocyanine pigments are produced in high yield and of good colour by using inorganic esters as catalysts. The esters are of formula $E(OR)_x$ (E = element from Grps. IV, V or VI of the Periodic Table of At. Wt. $\leqslant$ 28; R = Alk, aryl, alkaryl or mixtures thereof; x = valency of E). Particularly suitable are such derivatives of Ti or Zr. Thus, e.g. $TiCl_4$ is reacted with excess anhydr. CH_3OH in hexane whilst passing NH_3 gas through the system. At the end of reaction, filtration removes the NH_4Cl formed and the filtrate is distilled to remove hexane and excess CH_3OH. The residue ($Ti(OCH_3)_4$) (0·0466 mol.), trichlorobenzene (4·5 mol.), tetrachlorophthalic anhydride (0·2 mol.), urea (1·3 mol.), and $Cu(NO_3)_2 \cdot 3H_2O$ (0·056 mol.) are then heated at 175–85°C. and the product is recovered after cooling by filtration. After further washing etc. the dry pigment is obtained in 93·6% of theory yield. E.T.

Oil- and Spirit-soluble Phthalocyanine Derivatives
American Cyanamid Co. *USP* 2,823,205

The simple phthalocyanine pigments with their outstanding tinctorial and light-fastness properties are insol. in organic solvents and hence cannot be used for paints, inks, or the colouring of plastics. Products obtained by reacting phthalocyanine sulphonic acids with secondary amines are slightly alcohol-soluble but are still insol. in hydrocarbon oils. Reaction products of chloromethyl phthalocyanines and secondary amines have some oil-solubility but are expensive. The method of the present patent produces phthalocyanine derivatives which have greatly increased spirit- and oil-solubility; they are salts of formula—

(P = metal-free or metallised phthalocyanine nucleus; Q^+ = a quaternary ammonium ion; n = 1–8). The phthalocyanine starting material, viz. an *o*-carboxybenzamidomethyl substituted phthalocyanine (ref. *USP* 2,761,868) is produced by, e.g. adding to 100% H_2SO_4 (140) at 50–55°C. Cu phthalocyanine (10), 30% oleum (56·6), phthalimide (30·5), and *para*formaldehyde (11). Reaction is completed at 70–75°C. and the product is separated by drowning out and filtering to give an excellent yield of tetraphthalimidomethyl-Cu-phthalocyanine. Higher temperature of reaction introduces some SO_3H groups; varying the amounts of *para*formaldehyde and phthalimide raises the number of phthalimidomethyl groups introduced. By heating the reaction product in conc. H_2SO_4 at 100°C., the phthalimidomethyl groups are hydrolysed to *o*-carboxybenzamidomethyl groups. Formation of a quaternary ammonium salt follows, e.g. tetra-(*o*-carboxybenzamidomethyl)-Cu-phthalocyanine (6) containing approx. 0·4 SO_3H groups per mol. is convérted to its Na salt by stirring with a solution of NaOH (7) in water (580). At 75°C., a solution of hexadecyl benzyl dimethyl ammonium chloride (8) in water (700) is added. After stirring, the product is filtered, washed, and oven dried. E.T.

Alumina–Silicon Pigments
Columbia-Southern Chemical Corpn. *BP* 805,494

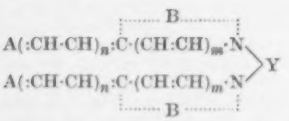
Finely divided aluminium silicate useful as a reinforcing pigment in rubber or for adding to paper pulp is obta ned by treating an alkaline earth metal silicate of average particle size $<$ 0·1 μ. in aqueous medium with enough of an aluminium salt to react with $\leqslant$ 20% of the alkali earth metal silicate. C.O.C.

Carotenoid-containing Oils for Colouring Foodstuffs
Harburger Oelwerke Brinckman & Mergell *BP* 804,685

An oily carotenoid-containing extract can be produced from palm oil without much decomposition of the carotenoids occurring during either production or storage of the extract if $\leqslant$ 10% by weight of a vegetable oil containing a substantial amount of a natural antioxidant is added to the raw or deacidified palm oil before or during its separation into solid carotenoid-poor and liquid or semi-liquid carotenoid-rich components. The preferred oils to be added are cottonseed oil, sunflower seed oil and germ oils, e.g. maize- or wheat-germ oil. C.O.C.

Photographic Sensitisers containing more than one *mero*Cyanine Dye Radical
ICI *BP* 804,670

Dyes of formula—



(m = 0 or 1; B = atoms to complete a 5- or 6-membered ring which may form part of a condensed ring system; Y = divalent polymethylene or xylylene radical linked to the N atoms through aliphatic C atoms; n = 0, 1 or 2; A = a 5- or 6-membered ring linked through a single C atom and containing a keto group on a C atom attached to the linking C atom), e.g. the red 3′′:3′′′-pentamethylene bis-[1-phenyl-3-ethyl-5-(5′′-methyl-2′′:3′′-dihydro-1′′:3′′:4′′-thiadiazolylidene-2′′)-ethylidene-2-thiohydantoin], obtained by condensing 3:3′-pentamethylene bis-[2(β-acetanilidovinyl)-5-methyl-1:3:4-thiadiazolinium bromide] with 1-phenyl-3-ethyl-2-thiohydantoin, are useful photographic sensitisers, as they are not readily removed from silver halide grains by colour formers containing Alk of > 7 C. They have no tendency to diffuse from layer to layer when used in multilayer material.
C.O.C.

Food Dye
Union Starch & Refining Co. *USP* 2,841,499

A product suitable for colouring powdered foods consists of caramel (C.I. Natural Brown 10) and/or certified dye, together with 0·25–1·0% (on the weight of the food) of water-soluble cellulose ethers. C.O.C.

Surface-coated Calcium Carbonate Pigments
Wyandotte Chemicals Corpn. *USP* 2,841,504

Finely divided calcium carbonate (C.I. 77220) is coated with both a C_{16-18} primary n-alkylamine or rosin amine and a C_{16-20} fatty acid, rosin acid or an ester or alkali metal salt of a rosin acid. The products are readily incorporated into rubber stocks. C.O.C.

Carbon Black (C.I. Pigment Black 7)
Phillips Petroleum Co. *BP* 806,092

In producing Furnace Black control of the quality of the product is exercised by analysing the effluent gas from the furnace and regulating the ratio of air to hydrocarbon supplied to the furnace according to the analysis.
C.O.C.

Finely Divided Silica (C.I. Pigment White 27)
ICI *BP* 805,491

Silica of particle size < 50 mμ. is obtained by adding a solution of an alkali metal silicate of strength ≮ 160 g./l. of SiO_2 to a solution of an ammonium salt with continuous stirring and keeping the mixture at > 60°c. C.O.C.

Titanium Dioxide (C.I. 77891)
DuP *BP* 805,572

A vaporised mixture of aluminium trihalide and titanium tetrahalide free from other metal halides is obtained by forming a fluidised bed of aluminium metal particles with excess of inert solid particles and passing into it a mixture of the vapour of a halogen and titanium halide. The bed is kept below the softening temperature of the aluminium particles but above the condensation temperature of the aluminium halide vapour formed. The mixture of halides is then subjected to vapour phase oxidation to produce titanium dioxide. C.O.C.

3:3′:5-Trihydroxydiphenyl as the Azoic Coupling Component in Diazotype Material (IX p. 132)

Mixtures of Cyanine Dyes and Cadmium or Zinc Chloride or Bromide as Supersensitisers (IX p. 132)

Fibrous Solid Silicon Monoxide— Reinforcing Pigment for Rubber (XIII p. 136)

Silicon Monoxide as a Reinforcing Pigment for Rubber (XIII p. 137)

Radiation Chemistry of Solutions. II— Dose Rate, Energy, and Temperature Dependence of a Leuco Triarylmethane Dosimeter Solution (XIV p. 137)

Metallised Azo Dyes for Staining Wood (XV p. 140)

V—PAINTS; ENAMELS; INKS

Pigment Wetting in Zein-Alcohol (Printing Ink) Varnishes
T. K. Maher and E. W. Janssen
Amer. Ink Maker, **36**, (7), 32–4, 61, 63 (1958): *Chem. Abs.*, **52**, 17748 (25 Oct. 1958)

Addition of a little of a surface active substance before milling the pigment into the vehicle improves the stability of zein flexographic inks against η increase. Development of thixotropy and gelation in the inks on standing is caused by poor wetting of the pigment by the varnish. In the trials TiO_2 (C.I. Pigment White 6), Chrome Yellow (C.I. Pigment Yellow 34), Iron Blue (C.I. Pigment Blue 27), Ba Lithol Red (C.I. Pigment Red 49), Ca Lithol Red (C.I. Pigment Red 49), and Carbon Black (C.I. Pigment Black 7) were milled in porcelain and steel ball mills with 20% and 30% solutions of zein in 90% aqueous alcohol in presence and in absence of a wetting agent. Viscosities were measured with a Brookfield viscometer immediately after grinding and then after storage. The Carbon Black inks were stable without addition of a wetting agent. The data are summarised in tables and photomicrographs.
C.O.C.

Colour in Writing and Copying
J. P. Gill
J.S.D.C., **74**, 739–745 (Nov. 1958)

A general summary of types of dyes and pigments used in inks (other than printing inks), pencils, typewriter ribbons, and carbon papers. An account of the properties necessary for the different applications is given, together with some particulars of the method of manufacture and use of typewriter ribbons and carbon papers, both for normal typing purposes and for spirit duplicating.
AUTHOR

PATENT

Erasable Ink for Ball Point Pens
Parker Pen Co. *BP* 805,362

Graphite (C.I. Pigment Black 10) of particle size < 7 μ., preferably ≯ 3–4 μ., is made into a 15–17% dispersion in a non-drying hydrocarbon oil in presence of an agent, e.g. asphalt or tar, which brings the viscosity of the ink to about 60 SAE at 25°c. The graphite may be replaced by a coloured pigment. C.O.C.

Coatings containing Tetrafluoroethylene Resin and Phenol-Aldehyde Resin (XIII p. 136)

VI—FIBRES; YARNS; FABRICS

Test Fungi in Textile Testing
H. Rämsch and C. Kühne
Faserforsch. und Textiltech., **9**, 441–445 (Oct. 1958)

Mould growth, breaking-load, and engraving tests are described and used to examine the action of bactericides and fungicides on cellulose textiles. Morphological characteristics of the most important test fungi are described and illustrated. W.R.M.

Textile Damage by Earwigs
S. Wolf
Faserforsch. und Textiltech., **9**, 445–449 (Oct. 1958)

Experimental tests show that damage of some textiles by earwigs may occur but is dependent on special conditions which include the mechanical strength of the textile material. W.R.M.

Constitution of Jute Hemicellulose—I
G. O. Aspinall and P. C. Das Gupta
J.C.S., 3627–3631 (Oct. 1958)

Partial hydrolysis of jute hemicellulose (I) yields (2-D-xylose 4-*O*-methyl-α-D-glucopyranosid)uronic acid, and hydrolysis of the methylated I affords 2:3:4-tri-*O*-methyl-D-xylose, 2:3-di-*O*-D-xylose, 3-*O*-methyl-D-xylose, and (3-*O*-methyl-2-D-xylose, 2:3:4-tri-*O*-methyl-α-D-glucopyranosid)uronic acid in the approx. mol. ratio 3:79:4:14. The methylated polysaccharide has a mol.wt. of 21000 ± 500 (degree of polymerisation, 123 ± 3). It is concluded that I is composed of chains of ca. 108 1:4-linked β-D-xylopyranose residues with approx. every seventh residue carrying a terminal 4-*O*-methyl-α-D-glucopyranosiduronic acid residue linked through position 2. A small degree of branching in the backbone of D-xylose residues is indicated.
H.H.H.

Light Damage to Disaccharides in the Presence of Titanium Dioxide Pigments
J. Schurz and E. Kienzl
Svensk. Papperstidning, **61**, 844–850 (15 Oct. 1958)
To obtain information on problems of light damage to cellulose, several disaccharides, alone or mixed with TiO_2, were irradiated with ultraviolet radiation and the resultant changes measured. The ultraviolet maximum was found to be dependent on pH and optical rotation was slightly altered. It would appear that the first stage of TiO_2-light damage of cellulose is oxidation of carbon atoms 2 and 3, leading to the formation of a double bond by enolisation. Molecular decomposition occurs in a second stage caused by further oxidation, leading to rupture of the ring with the formation of carboxyl groups and, finally, breaking of the –C–O–C bond and, in the case of cellulose, to a break-down of the macromolecule. The presence of inorganic pigments may be a contributing, if not even the sole factor, of indirect light damage. R.A.

Spaces in a Uniform Random Suspension of Fibres
A. G. Ogston
Trans. Faraday Soc., **54**, 1754–1757 (Nov. 1958)
The distribution of spaces in a random network of straight fibres is considered, such as might be occupied by a spherical object placed within the network or might allow free rotation to an elongated one. The approximate treatment of a network of flexible fibres is briefly discussed. W.R.M.

Empirical Relations of Crease Recovery and Time
P. R. Wilkinson and H. E. Stanley
Text. Research J., **28**, 669–673 (Aug. 1958)
A technique for measuring short-time crease recovery is described. Data from this method and from a standard method are used to establish several empirical relationships between crease recovery and time which give linear plots and which permit extrapolation to very short and very long times. The relationships lead to several new parameters for characterising fabric recovery. These are very short-time crease recovery, rate of delayed recovery, and recovery after infinite time. These parameters depend primarily on fibre type and to a much lesser extent on fabric construction. S.B.D.

Preparation and Properties of Partially Benzylated Cotton
E. Klein, D. J. Stanonis, P. Harbrink, and R. J. Berni
Text. Research J., **28**, 659–668 (Aug. 1958)
Ternary systems of alcohol, water, and sodium hydroxide can be used in a pretreatment bath or added in fixed quantities to cotton prior to etherifying with benzyl chloride. The evidence obtained supports an hypothesis that increased reagent solubility, coupled with control of the cellulose–soda cellulose equilibrium, can lead to essentially homogeneous cotton cellulose modifications. In addition, the experiments showed that interesting fibrous cellulose ethers can be prepared. These ethers retain many of the desirable mechanical properties of the original cotton and show increases in elastic recovery, decreased permanent set, substantivity to disperse dyes, microbiological resistance, and a high degree of thermoplasticity. S.B.D.

Nature of Permanent Set in Keratin Fibres
M. Feughelman, A. R. Haly, and T. W. Mitchell
Text. Research J., **28**, 655–659 (Aug. 1958)
The permanent set of keratin fibres in water is mainly due to strong polar linkages formed during the setting process. These linkages are identified with the bonds broken in the second stage of supercontraction of a fibre in a conc. LiBr soln. The bonds are probably formed by groups of hydrogen bonds which are closely associated in the keratin structure with a tyrosine residue. S.B.D.

Maximum Alkali-combining Capacity of Wool
J. R. McPhee
Text. Research J., **28**, 714–716 (Aug. 1958)
Using alkaline soln. saturated with certain inorganic salts, usually sodium chloride, enables the max. alkali-combining capacity of the wool to be found with much less degradation and loss of weight than is normal at high pH. The mean value obtained in this way agrees reasonably well with the average value calculated from the sums of the arginine, lysine, α-amino group, tyrosine, and histidine contents of wool, being 11·8 and 12·2 g-equiv. $OH^-/10^4$ g. dry wool respectively. S.B.D.

Cause of Wool Felting
O. K. Dobozy
Text. Research J., **28**, 717–719 (Aug. 1958)
Placing wool fibres in water causes heterogeneous swelling of the cortical components. The less highly orientated areas swell more along the fibre axis than do the more highly orientated areas and at first cause the fibre to lose its natural crimpiness. With further swelling, certain points form new curvatures in the opposite direction to the crimp, giving rise to large fibre movements and entanglements. The felting power of a wool should, therefore, be expressed in terms of three parameters — the hysteresis of the extension and contraction curve, the directional friction of the fibre, and the tendency of the fibre to entangle. This latter parameter may be expressed as the length of the fibre divided by the radius of the sphere enclosing the entangled fibre. S.B.D.

Electron-microscope Studies of the Chemical Reactivity in Keratin Cuticle
J. Sikorski and W. S. Simpson
Nature, **182**, 1235–1236 (1 Nov. 1958)
Keratin fibres are treated with mercury or lead salts which form coloured metal sulphides in keratin. Examination of cross sections shows that with lead salts the differentiation of layers in the cuticle is more clearly shown than when osmic acid is used. W.R.M.

Table of Synthetic Fibres
P.-A. Koch
Z. ges. Textilind., **60**, (18), 797–801 (Sept. 1958)

Effects of Different Fibre Contours with and without Internal Hollow Spaces
H. Böhringer and F. Bolland
Faserforsch. und Textiltech., **9**, 405–416 (Oct. 1958)
The advantages of fibres with definite contours or profiles and with internal hollow spaces are indicated. Fibre cross-sections obtained using different nozzle apertures are illustrated and their textile physical properties discussed. The effects on spinning, weaving, knitting, and finishing and on the fabric characteristics are studied and discussed. The value of such fibres is demonstrated by the results of wear and tear tests. W.R.M.

Dielectric Properties of Solid Polymers
L. de Brouckere and G. Offergeld
J. Polymer Sci., **30**, 105–118 (July 1958)

Cross-linking and Porosity of Macromolecules
J. A. Mikes
J. Polymer Sci., **30**, 615–623 (July 1958)
With inert additives, chain-forming and cross-linking monomers polymerised to give copolymers of which the swelling, hydration, diffusion constant, inner surface, and porosity are anomalous. P.T.S.

Dielectric Properties and Polarisation of Polymers
G. P. Mikhailov
J. Polymer Sci., **30**, 605–614 (July 1958)

Cold-drawing of Glass-like and Crystalline Polymers
J. S. Lazurkin
J. Polymer Sci., **30**, 595–604 (July 1958)
Intermolecular bonds are broken during the cold-drawing of both glass-like and crystalline polymers. The activation energy of the molecular rearrangement is decreased with increasing stress. The mechanical relaxation properties of a polymer change on orientation. P.T.S.

Structure and Phase State of Polymers
V. A. Kargin
J. Polymer Sci., **30**, 247–258 (July 1958)
X-Ray and electron-microscope studies of thin films of crystallisable polymers above and below the crystallisation temperature has shown that a fair mutual ordering of chain molecules can occur even in the amorphous state, and that crystallisation is but the last step in the process of mutual ordering of chain molecules. P.T.S.

Isothermal Volume Contraction of Amorphous Polymers
A. J. Kovacs
J. Polymer Sci., **30**, 131–147 (July 1958)
An amorphous polymer is quenched from a temperature above the glass transition temperature to one below it. The results lead to a precise kinetic definition of the glass transition temperature. P.T.S.

Effect of Temperature on the Strength and Elongation of Fibres
A. V. Motorina and A. A. Konkin
Tekstil. prom., **18**, 18–20 (July 1958)

The change in strength and elongation of fibres from Capron, Anid, Enant (polyaminoheptanoic acid), Nitron, polyester, and viscose rayon have been studied at 140°c. and in the range +20 to −50°c. The mechanical properties of polyester and Nitron fibres were least affected by change in temp. With decreasing temp. all fibres show an increase in strength and a decrease in elongation, these changes being reversible. G.J.K.

Polymeric Cyclic Amides from Polycaprolactam
M. Rothe
J. Polymer Sci., **30**, 227–238 (July 1958)

In the polymerisation of ε-caprolactam about 3% cyclic oligomers are formed. Because of their similar properties and the absence of end groups these different cyclic polymers are not easily distinguishable and this paper describes the preparation of pure reference substances. Heating in the presence of water causes an equilibrium to be set up between all the possible cyclic and linear polymers. P.T.S.

Mechanism of the Polymerisation of ε-Caprolactam
I— Polymerisation initiated by ε-Aminocaproic Acid
C. A. Kruissink, G. M. van der Want, and A. J. Staverman
J. Polymer Sci., **30**, 67–80 (July 1958)

A "differential" method, in contrast to the usual "integral" method, is used to test proposed reaction mechanisms against the experimental results. The velocities of reactions are obtained by graphical integration of curves representing the amounts of reagents *versus* time. It is claimed that the method is simpler and yields better results.

II— Polymerisation in Presence of Water
P. H. Hermans, D. Heikens, and P. F. van Velden
Ibid., 81–104

The addition of lactam on to the end-group of a linear polymer molecule is catalysed by another end-group. At the start the hydrolysis of lactam by water is uncatalysed, but later the hydrolysis catalysed by end-groups is more important. Cyclic oligomers are produced by transamidation reactions rather than by cyclic condensation. P.T.S.

Alkaline Polymerisation of Caprolactam. IV— Equilibrium and Degradation with Alkaline Polymers of Caprolactam
J. Králíček and J. Šebenda
J. Polymer Sci., **30**, 493–499 (July 1958)

The sodium salt of caprolactam was used as a redistribution catalyst with samples of alkali-polymerised caprolactam. The equilibrium value of the intrinsic viscosity after redistribution of molecular weights depends on the catalyst concentration and the temperature at which the redistribution is carried out. The concentration of basic groups remains constant during the redistribution. P.T.S.

Alkali-catalysed Polymerisation of Caprolactam
J. Saunders
J. Polymer Sci., **30**, 479–492 (July 1958)

The rapid, base-catalysed addition polymerisation of caprolactam gives a less predictable molecular weight distribution in the polymer than the slower polycondensation process. If water, 6,6-nylon salt, aminocaproic acid, acetic acid, or *n*-butyl alcohol are introduced at a late stage into the base-catalysed polymerisation, redistribution of molecular weight occurs by hydrolysis or chain interchange reactions and a reproducible molecular weight distribution is obtained. The polycaproamide after washing with water contained an excess of free amine end-groups over carboxyl end-groups. No chain branching was detectable by infrared or viscosity measurements. P.T.S.

Polyester Formation by Re-esterification
W. Griehl and G. Schnock
J. Polymer Sci., **30**, 413–422 (July 1958)

Polyester formation from "precondensed" polyethylene terephthalate was followed by viscosity measurements and found to be a first-order reaction. P.T.S.

Re-equilibration of Polycaproamide
S. Smith
J. Polymer Sci., **30**, 459–478 (July 1958)

The polymerisation of caprolactam and the depolymerisation of polycaproamide at equilibrium lead to an identical mixture of linear polymer, caprolactam monomer, and cyclic oligomers. Hydrolysis and inter- and intramolecular interchange reactions are responsible for depolymerisation— its rate is controlled by the concentrations of water and amine and carboxylic end-groups. In the discussion, amide–amide interchain exchange reactions are mentioned. P.T.S.

Mechanism of Growth of Macromolecules of Polyethylene Terephthalate during the Polycondensation
E. Turska, T. Skwarski, and S. Szapiro
J. Polymer Sci., **30**, 391–398 (July 1958)

A formula is derived which permits the calculation of the differential molecular weight distribution curve as a function of the intrinsic viscosity of the unfractionated polymer sample. P.T.S.

Reclamation of Fibres from Rags. III— Action of Hydrazine on Terylene
A. Filson and J. B. Speakman
J.S.D.C., **74**, 762–765 (Nov. 1958)

The extent to which Terylene is attacked by 2% solutions of hydrazine in various alcohols at 100°c. is closely, but inversely, related to the heats of mixing of hydrazine and the alcohols. With glycerol and ethylene glycol the heat of mixing is high, and it has been shown that ethylene glycol forms a well defined 1:1 complex (m.p. 9·2°c.) with hydrazine. Dilute solutions of hydrazine in these solvents contain little free hydrazine, and it is not surprising, therefore, that they cause little degradation of Terylene in 40 min. at 100°c. Conversely, the most rapid destruction of Terylene is brought about by solutions of hydrazine in less reactive solvents, such as toluene and *iso*amyl benzyl ether. Such solutions have the additional merit of discriminating sharply between wool and Terylene, thus allowing the former to be recovered from mixtures for re-use. AUTHORS

PATENTS

Improving the Dyeability of Hydrophobic Polymers
Dow Chemical Co. *BP* 805,513

Impregnation of a hydrophobic polymer, particularly acrylonitrile, with vinylpyrrolidone, vinylbenzene-4-sulphonic acid, *N*-vinyl-*N*-methyl(or ethyl)formamide, *N*-vinyl-*N*-methyl(or ethyl)acetamide, *N*-vinylvalerolactam, *N*-vinylcaprolactam acrylamide or vinylpyridine, and then polymerising *in situ* yields products of improved affinity for dyes. The dyeings obtained on such products are well penetrated and of markedly improved wet fastness to those obtained on the untreated polymer. C.O.C.

Treating Polyethylene Terephthalate Fibres with Chlorinated Aliphatic Hydrocarbons
Vereinigte Glanzstoff-Fabriken *BP* 805,525

When polyethylene terephthalate fibres are treated for a short time, preferably up to 10 min., with a chlorinated aliphatic hydrocarbon containing an H atom attached to C, e.g. chloroform, trichloroethylene, dichlorobutane, tetrachlorobutane, *s*-dichloroethylene, 1:2-dichloroethane, or pentachloroethane at < 120°c., preferably at 60–80°c., the fibres shrink, stiffen, and their surface acquires a slight roughness which improves the adhesion of size and lubricants to them. The process may be used to crimp fibres and threads of polyethylene terephthalate. C.O.C.

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Bleaching Cotton with Nascent Peracetic Acid
K. Prett
Melliand Textilber., **39**, 999–1001 (Sept. 1958)

The drawbacks of kier-boiling and alkaline bleaching are summarised. Dilute acid hydrogen peroxide almost quantitatively converted to nascent peracetic acid in the bleaching liquor by acetic anhydride with a catalyst does not affect most vat and azoic colours, unlike acid chlorite, and is both more efficient and pleasant than technical peracetic acid. S.M.J.

Non-silicate Stabilisation of Hydrogen Peroxide Bleach Solutions
D. M. Cates and W. H. Cranor
Text. Research J., **28**, 708–713 (Aug. 1958)

Alkaline soln. of hydrogen peroxide are stabilised by addition of calcium chloride and disodium phosphate. Evidence is given that stabilisation may be due to the formation of calcium hydrogen phosphate as ppt. Adding disodium diphosphate to liquors containing fixed amounts of hydrogen peroxide, calcium chloride, and sodium hydroxide, causes the pH of the soln. to rise to a max. then fall to a buffered level, the decomposition curve of the peroxide following the same pattern. The final soln. obtained this way is much more stable than those maintained at the same or lower pH in the absence of calcium hydrogen phosphate. S.B.D.

Bleaching Packages of Cotton Yarn with Peroxides and Sodium Hypochlorite
J. Cegarra Sanchez and L. V. Albert
Ing. textil (Barcelona), **25**, 25–49 (1958): *Chem. Abs.*, **52**, 17721 (25 Oct. 1958)

Bobbins bearing 800–900 g. of cotton yarn had bleach liquor circulated through them in a closed system under pressure, the liquor circulating from the inside to the outside of the packages. pH was kept at 10·5–10·8 by use of Na_2SiO_3, Na_2CO_3, or NaOH. Using 0·3% aq. H_2O_2 (1 mol.) native (Spanish) cotton was bleached suitably for white goods in 2 hr. at 120°c. but not at 85°c. At 110°c. a borderline bleach was obtained. By doubling the amount of H_2O_2 used a bleach was obtained at 85°c. which was suitable for goods to be dyed. Variation in excess static pressure in the range 1–3 kg./sq.cm., controlled by introducing compressed air into the expansion chamber, had no detectable effect on the resultant bleach, although increased pressure reduced the rate of decomposition of H_2O_2. Small loss in yarn tenacity occurred at 85°c. together with very slight increase in Cu-reducing groups and no detectable change in degree of polymerisation of the cellulose. At 110° and 120°c. the mol.wt. of the cellulose calculated from the degree of polymerisation decreased by about half and greater increase in Cu-reducing groups occurred. The Methylene Blue number remained constant. Coarser yarns absorbed O more rapidly because the packages were more permeable to the liquor, while the finer yarns loss more in strength. Less pigmented American cotton was more easily bleached than Spanish cotton. Na_2O_2 gave as an efficient bleach as H_2O_2 but the bleached yarn was rougher. NaClO was less effective than the peroxides, but cost only a third as much. C.O.C.

Laboratory Studies of Bleaching Flax and Hemp with Hydrogen Peroxide
L. Merlo, G. Vitelli, and A. Arbo
Ann. Sci. Textiles belges, **4** (2), 11–18 (1956): *Chem. Abs.*, **52**, 17721 (25 Oct. 1958)

Tests were carried out by (1) treating the material for 60 min. at 90–95°c. in Na_2SO_3 (5 g./l.), Na_3PO_4 (5), $NaHSO_3$ (3), and wetting agent (3), liquor ratio 7 : 1, and then bleaching for 75 min. at 85–90°c. in 130 vol. H_2O_2 (13) and Na silicate (15); (2) treating the material for 90 min. at 100°c. in an aqueous solution containing 1% NaOH and 1% wetting agent, liquor ratio 10 : 1, and then bleaching for 60 min. at room temperature in a solution containing 2·5 g./l. active Cl and 60 min. at 80°c. in 1% aqueous chlorite brought to pH 3 with active acid; (3) treating for 150 min. at 90–95°c. in 5·5% aq. Na_2CO_3 containing 1% wetting agent, and then bleaching for 60 min. at room temperature in an aqueous solution containing 12 g./l. active Cl. Treatment for 20 min. in 1 g./l. aq. $NaHSO_3$ is given as an antichlor. The first method does not affect the breaking strength of flax, whereas the second and third methods significantly decrease it. The cuprammonium viscosity of flax is decreased from 45 to 41·37 by the first treatment, to 26·50 by the second, and to 11·73 by the third. The colour of flax bleached by the first method is superior to that obtained by the other two. Similar results hold good for hemp. C.O.C.

PATENT

Bleaching of Vegetable Fibres
DuP *BP* 805,749

The goods are moistened with an aqueous solution of a peroxy compound at $< 275°F$. under conditions which prevent them drying out. Thus thoroughly prepared muslin was impregnated with its own weight of a liquor containing 0·9% H_2O_2, 1% 42° Bé. Na silicate, and NaOH to bring to pH 10·7, and heated for 5 sec. in an autoclave at 300°F. This yielded an excellent bleach. C.O.C.

VIII— DYEING

A Scottish Dyehouse in the Seventeenth Century
R. A. Peel
Dyer, **119**, 609, 610 (25 April); 901–903 (20 June 1958); **120**, 167, 168 (1 Aug.); 379–381 (12 Sept.); 640–642 (24 Oct.); 791–794 (21 Nov. 1958)

Minutes relating to wool dyeing are reproduced from the records of the Scottish Cloth Manufactory at New Mills, near Haddington, covering the period 1681–1703. Explanatory notes are added. C.J.W.H.

Continuous Dyeing of Wool with Milling Acid Dyes
D. R. Lemin
J.S.D.C., **74**, 746–756 (Nov. 1958)

It has been shown that loose wool may be successfully dyed continuously with milling acid dyes in very short times at 100°c. in normal scouring machines, thus eliminating normal dyeing machinery. The method is restricted commercially to pale and medium depths. A pad–steam method has been developed for dyeing wool pile fabrics continuously. It has been found that it is not possible to dye wool piece goods satisfactorily by continuous pad–steam methods, owing to the hydrophobic character of the wool and the difficulty of eliminating skitteriness from the dyeings. AUTHOR

Dyeing with Variamine Blue B Salt (C.I. Azoic Diazo Component 35) and Variamine Blue RT Salt (C.I. Azoic Diazo Component 22)
K. Janiszewski
Prace Inst. Włókiennictwa, **7** (2), 1–20 (1957): *Chem. Abs.*, **52**, 17719 (25 Oct. 1958)

When coupling with 10–20 g./l. of the above a reduction of 20% of the amount necessary can be effected by using $ZnSO_4$ 0·2 g./l. as buffer and Oleol 18 (octadecyl alcohol condensed with a chain containing 18 $-OC_2H_4-$ groups) 0·3 g./l. as assistants in the bath. Coupling time of 3–5 sec. and pH 7–8 are the most suitable. Use of $C_{18}H_{37}(OC_2H_4)_{15-20}OC_2H_4OH$ or Diazopon 15 as assistants or Na acetate + acetic acid, formic acid + Na formate, disodium hydrogen phosphate, $NaHCO_3$, or $MgSO_4$ as buffers had no beneficial effect. C.O.C.

Fading and Tendering Activity in Anthraquinonoid Vat Dyes
C. J. Cooper and H. I. Stonehill
IV— Polarographic Oxidation of *leuco*-Dyes
J.C.S., 3341–3352 (Oct. 1958)

Polarographic oxidation of the *leuco*-soln. in 48% aq. ethanol of 11 anthraquinonoid vat dyes (some of which exhibit fading and tendering activity) and one related quinone at 25°c. and pH 11·15–12·88, gave the following results: most of them give absorption waves; Caledon Gold Orange G, Cibanone Golden Yellow GK, and Caledon Blue RC, afford a single diffusion wave corresponding to one-step oxidation of quinol to quinone; Algol Yellow WG, Anthranone, Caledon Brilliant Orange 4RN, and Caledon Orange 2RTS give, in addition, another of different height, at more positive potentials, ascribed to bivalent oxidation of the tautomeric oxanthranol; Caledon Red 5G and Caledon Red X5BS give two such additional waves ascribed to bivalent oxidations of some anthrone and anthranol formed by over-reduction of the oxanthranol during vatting; and Cibanone Brilliant Orange GK, 6R, and Cibanone Golden Yellow RK, give two diffusion waves of approx. equal height, probably due to bivalent oxidations of quinol and oxanthrol. Comparison with potentiometric data indicates that polarography is incapable of revealing the formation of relatively stable semiquinones by many of these dyes. There is no obvious relation between half-wave potentials for quinol oxidation and tendering activity, although more negative values tend to be associated with fading. Intense irradiation of the *leuco*-soln. in the region of the dropping-mercury electrode with visible light, even in the presence of potential mediators, or with X-rays, is without effect on the polarograms; this contrasts with the effect during potentiometric titration of tendering-active dyes.

V— Photo-dehalogenation and -oxidation of *leuco*-Solutions

Ibid., 3353–3361

leuco-Solutions of the halogenated dyes Cibanone Brilliant Orange GK and Gold Yellow RK, and Caledon Brilliant Orange 6R, Orange 2RTS, and Brilliant Orange 4RN undergo rapid dehalogenation when exposed to visible light. In the presence of excess of dithionite, dehalogenation is complete, but, in catalytically prepared *leuco*-soln. with no excess of reductant, it proceeds to the extent of statistically one halogen atom per mol., which releases sufficient oxidant to oxidise completely and thus ppt. all the *leuco*-dye present. Thus, a dye containing n halogen atoms per mol. can undergo n successive reduction-irradiation-pptn. cycles. Potentiometric determinations show that not all the halogen liberated appears as halide ion; some is probably lost by adsorption on pptd. dye. Examination of the irradiation products by chromatography, etc., confirms the dehalogenation, and indicates that no monohalogenated product is formed from either di- or tri-halogenated dyes. The occasional production, under severe reducing conditions, of a *leuco*-soln. which was not rapidly pptd. on irradiation, an effect enhanced by pyridine, is ascribed to the formation of tautomeric oxanthranol. The spectral region responsible for photo-dehalogenation is located as approx. 450–650 mμ. for all five dyes.

VI— Coulometry of the Polarographic Oxidation of *leuco*-Dyes

Ibid., 3362–3367

The number of electrons involved in the polarographic oxidation of *leuco*-soln. of the eleven dyes of IV, anthanthrone, and Caledon Blue RSN, are determined micropolarographically, and found to be 2 for all the cpd. except the five which are halogenated and undergo photo-dehalogenation-oxidation in *leuco*-soln.; for these, the apparent number of electrons is one. By conventional and differential absorption spectroscopy, the products of the exhaustive polarographic oxidation of the dihalogenated photo-active *leuco*-dyes are shown to contain ca. 25 moles % of the analogous unhalogenated dye. Both this partial dehalogenation and the low electron loss during electro-oxidation are accounted for by a tentative reaction scheme, in which a *leuco*-dye mol. adsorbed on the mercury-drop anode is dehalogenated by the joint action of mercury and water, either during or after electro-oxidation, yielding mercury halide and a hydroxyl radical, which then oxidise another *leuco*-dye mol. H.H.H.

Constitution of Remazol (FH) Dyes

H. Zimmermann

Melliand Textilber., **39**, 1026–1027 (Sept. 1958)

Remazol (FH) dyes such as dye $-SO_2\cdot CH_2\cdot CH_2\cdot O\cdot SO_3H$ react in alkaline medium with cellulose via vinyl residues giving cellulose ethers like cellulose$-O\cdot CH_2\cdot CH_2\cdot SO_2-$dye; hydrolytic side-reactions probably yield hydroxyethyl sulphones of little affinity. S.M.J.

Effect of Afterchrome and Vat Dyeing on Manufacture and Wear Characteristics of Wool

W. Hausmann

Melliand Textilber., **39**, 900–905 (Aug.), 1013–1017 (Sept. 1958)

Loose wool is dyed with afterchrome and vat dyes, spun, woven, and converted into garments. Part of the vat-dyed wool is aftertreated with an antibacterial agent (Eulan FL), part of the chromed wool is spun and woven differently. Six months' wearing trials of 80 suits dry-cleaned weekly show no significant differences, whereas manufacturing and mechanical properties appear superior for the vat-dyed wool. For this, dyeing is shorter, steam-consumption lower, though dyestuff costs are higher; in spinning yield, yarn strength and extension are higher; breakages in spinning and weaving are lower; less time is required in milling, but more in washing; wet and dry tear strengths, extensions (less markedly) and bursting strengths before and after abrasion are higher, though abrasion lowers the latter more than for chrome-dyed wool. The antibacterial treatment has little effect on the above properties, whilst different spinning and weaving improves the chrome-dyed wool, albeit not to the level of the vat-dyed wool. One year's weathering does not lower abrasion resistance; tear strength of vat-dyed cloth, though lowered more, is still better. S.M.J.

Reaction of Cellulose Fibres with Reactive Dyes and the Influence of Fibre Structure

K. Schwertassek

Faserforsch. und Textiltech., **9**, 321–329 (Aug. 1958)

Swelling and solution in sulphuric acid is used to study the reaction of cellulose fibres with cold-dyeing Procion dyes. Diminished solubility suggests cross-linking. Variations in structure may also be shown by this method. No effect on solubility was observed when hot dyeing Procion and Cibacron dyes were used. Sorption of iodine suggests blocking but not cross-linking. W.R.M.

Application of Procion (ICI) Reactive Dyes by Continuous Processes

R. A. Brindley and I. D. Rattee

Melliand Textilber., **39**, 1017–1022 (Sept. 1958)

The pad(with $NaHCO_3$)-dry system can employ (1) hot-flues, (2) steam-heated drying cylinders, or (3) fluid beds. In (1) the goods are cool ($< 60°$c. for air temp. 110°c.) until moisture content falls below saturation value— rapidly with rapid air displacement— when fixation, requiring 2–4 min., substantially ceases owing to insolubilisation of dye, especially in deeper colours. This can be counteracted by altering drying conditions, better by introducing some steam which reduces drying rate by ca. 20% and increases rate of fibre–dye reaction by 20–60%, best by incorporating solvents such as urea in the pad-liquor, which increase fixation by lowering rate of evaporation, raising temp. of goods, and delaying pptn., but also favour migration. (2) provide more rapid heat transfer and therefore higher wet goods temp. and faster (60–90 sec.) and more extensive fixation. Light goods react more rapidly but less extensively than heavy or raised fabrics. Passing fabric between cylinder and felt increases reaction rate and decreases migration rate. (3) dry and fix in 10–12 sec. The pad–steam process, best with high expressions, fixes pale colours in 10–30 sec. Intermediate drying enables greater depths and Procion H brands also to be fixed in 3 min. Padding first with dye, then with NaOH in brine, and steaming, if performed wet on wet, is restricted to very short caustic pad liquors, as in the Standfast molten metal machine, or to pale colours, because the dye bleeding into the brine, hydrolysed only to the monohydroxymonochlorotriazinyl derivative, can still react and cause ending; if wet on dry, can be used for all depths of cold-dyeing or "H" Procions, fixed by $\not< 15$ or $\not< 60$ sec. steaming. Fixation characteristics of the Procion range under all these conditions are illustrated in 10 tables and 4 graphs. Also discussed are removal of unfixed dye, combined dyeing and resin finishing, and the dyeing of loose viscose rayon. S.M.J.

Dyeing Furs with Oxidation Dyes

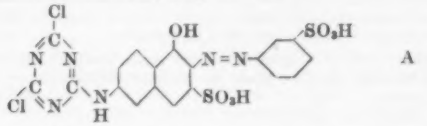
F. Brugger and L. Páris *Kožařství*, **7**, 354–7 (1957): *Chem. Abs.*, **52**, 19208 (10 Nov. 1958)

The conditions for the oxidation of *p*-phenylenediamine (C.I. 76060) by H_2O_2 in presence of $K_2Cr_2O_7$ were studied by determining the weight of insoluble oxidation products formed. Maximum catalytic effect was obtained with 0·05 oxidative equivalents of bichrome per C.I. 76060. Aqueous 3% bichrome was added to a solution of C.I. 76060 in a thermostat. Half the H_2O_2 (3% soln.) was added after 1 hr. and the other half after 2 hr. The temperature was maintained for a further 2 hr., after which the mixture was cooled overnight and the precipitate weighed. In presence of bichrome the maximum precipitate was formed with 3 equivalents of H_2O_2 (less precipitate with more H_2O_2), varying the temperature between 30–60°c. had no effect. In absence of bichrome, the precipitate increased with temperature, and the maximum precipitate obtained with 6 equivalents of H_2O_2 was only 25–50% of that obtained in presence of bichrome. Chromic salts have the same effect as bichrome, a significant point in the dyeing of lambskins tanned with chromic salts. In the absence of bichrome, addition of NH_4OH yields deeper dyeings, but the reverse is true in presence of bichrome. Dispersion of the precipitate is greater in presence of NH_4OH. When phenol is added to C.I. 76060, the weight of the precipitate increases but is not proportional to the weight of phenol added. The oxidation products of C.I. 76060 plus phenol contain less N, are less soluble and therefore more suitable as dyes than the oxidation products of C.I. 76060 alone. Pyrocatechol and resorcinol behave similarly to phenol but offer no advantages. C.O.C.

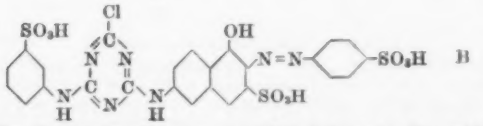
Reactive Dyes on Wool

R. Casty *S.V.F. Fachorgan*, **13**, 586–597 (Oct. 1958)

Reactive dyes consist mainly of dichlorotriazinyl (A) and monochlorotriazinyl (B) systems—



A



B

The former (much more reactive) belong to the cold-dyeing brands; the latter (less reactive) constitute the hot-dyeing class. Due to the reactivity of –Cl atoms in the triazinyl systems covalent bonds are formed between the dye and the substrate. The resulting dyeings are very fast to wet treatments. The fixation of reactive dyes on cellulose requires the addition of alkali. They are best applied to wool under mildly acid conditions. To avoid "skittery" dyeing the addition of certain cation-active assistants such as Neovadin AN (Ciba) is recommended. From a series of photomicrographs of fibre cross-sections, taken after 10, 20, 30, 40, 50, and 90 min. of dyeing (in the presence of Neovadin AN) it is apparent that although the dyebath was exhausted almost 100% after the first 30 min. of dyeing, the diffusion of the dye into the fibres did not commence until 10 min. later and was completed after 90 min. of total dyeing time (60 min. at the boil). It is possible that the anionic dye and cationic Neovadin AN form a complex which is initially deposited as a film round the wool fibres. When the bath is brought to the boil diffusion of the dye (or the complex) proceeds uniformly. Resulting dyeings are fast to rubbing, washing, potting, and light. The highest degree of fastness is obtained when surplus dye, which did not combine with wool, is removed by raising the pH of the bath to about 8 with ammonia or hexamethylene tetramine. Fastness properties on chlorinated wool are said to be as good as those on untreated wool. Reactive dyes are also of interest for Vigoureux printing of wool. L.A.T.

Dyeing Wool–Polyester Blends. IV— Effect of Dyeing Temperature

A. Würz *Melliand Textilber.*, **39**, 1023–1026 (Sept. 1958)

Fabrics were dyed at 94–100°c. for 1–8 hr. using a carrier— Palatinit M (BASF)— and yarns at 105–120°c. for 1 hr. using a dispersing agent— Uniperol W (BASF)— with 30 Palanil (BASF) disperse dyes. These differ appreciably in temp. sensitivity. Temperatures of 115°c. and 120°c. often give better results than carrier dyeing, unlike 105°c. and 110°c. Relative depths on both fibres are extensively tabulated. 7 of the dyes are recommended for 94°c., an additional 5 for 100°c. S.M.J.

Chemical Treatment of Polyamide Fibres with Pyridine Derivatives

S. Chrzczonowicz and B. Ostaszewski

I— The Use of Pyridine Derivatives as Assistants in Dyeing of Steelon (6-Nylon)

Zesz. Nauk. Politech. Lodz., No. 22 (*Chemia* 7), 47–56 (1958)

The possibility of the application of certain pyridine derivatives as assistants in the dyeing of Steelon, using acid and chrome dyes, has been examined. It was found that with acid dyes, pretreatment of the fibres with 2-(β-hydroxyethyl)-pyridine increased the depth of shade while at the same time causing some yellowing and precipitation of the dye in the dyebath. Poly-2-vinyl pyridine sulphate, used as a swelling agent with acid dyes, produced more uniform and intense dyeings on Steelon than on wool. The washing fastness was also improved. Quaternary derivatives of poly-2-vinyl pyridine used as surface-active agents in the dyeing with acid and chrome dyes produced improvement in fastness to washing to approximately the same degree as with Neolan Salt A (Ciba). The tensile properties of Steelon filaments remained unaltered as a result of the above pretreatments.

II— The Use of Quaternary Compounds Based on Poly-2-Vinyl Pyridine as Antistatic Agents

Ibid., 57–68

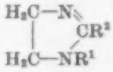
Preparations based on poly-2-vinyl pyridinium dimethyl sulphate were found to act as very efficient antistatic agents for continuous filament Steelon. Their effectiveness was increased by the addition of certain hygroscopic compounds such as glycerine, 2-(β-hydroxyethyl)pyridine, or electrolytes, e.g. NaCl, but not $CaCl_2$ due to formation of insoluble precipitates with Ca^{++} ions. The above preparations were stable over a wide range of temperatures and the treated yarn had long storage life under conditions which did not require strict control of temperature and humidity. L.A.T.

PATENTS

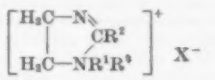
Level Dyeing of Nylon with Anionic Dyes in Presence of Substituted Imidazoline Compounds

Chemstrand Corpn. *BP* 806,026

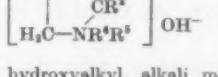
Level dyeings are obtained on nylon with direct, acid, premetallised acid, and afterchrome dyes applied from an aqueous acidic bath in presence of either a salt of an imidazoline of formula—



(R^1 = Alk, aralkyl, phenyl, hydroxyalkyl, or carboxyalkyl; R^2 = open aliphatic chain of 10–18 C), e.g. 1-butyl-2-hexadecylimidazoline, or a quaternary imidazolinium compound of formula—



(R^3 = same or different from R^1; X = Hal), e.g. 1:1-di-(2-hydroxyethyl)-2-heptadecylimidazolinium chloride, or a quaternary imidazolinium compound of formula—



(R^4 and R^5 = hydroxyalkyl, alkali metal-substituted hydroxyalkyl and alkali metal-substituted carboxyalkyl), e.g. disodium salt of 2-undecyl-1-(2-hydroxyethyl)-1-(carboxymethyl)-imidazolinium hydroxide. C.O.C.

Colouring Mineral Wool

American Rock Wool Corpn. *USP* 2,841,858

The fibres are coloured by adding a colorant, e.g. yellow iron oxide (C.I. Pigment Yellow 42), and a surface-active agent, to the annealing oil sprayed into the fibres at the entrance to the blow chamber in which they are collected. The resultant coloration is fast to rubbing and leaching. C.O.C.

Phthalogens (FBy) and Phthalocyanines (IV p. 121)

Polyamine Oxidation Dyes for Animal Fibres (IV p. 125)

Improving the Dyeability of Hydrophobic Polymers (VI p. 128)

Pigment Printing or Padding (IX p. 132)

IX— PRINTING

Recent History in Textile Printing

P. J. Wood *Amer. Dyestuff Rep.*, **47**, 755–756 (3 Nov. 1958)

Dyes for Photography— a Century of Progress

L. G. S. Brooker and P. W. Vittum *Proc. Perkin Centennial* (Lowell, Mass.: AATCC. 1958), pp. 369–408: *J. Photographic Science*, **5**, 71–88 (1957): J.S.D.C., **74**, 765–767 (Nov. 1958)

Discharge Printing Trevira Polyester Fibre
K. Roth

Melliand Textilber., **39**, 1027–1030 (Sept. 1958)

Solution of dyes in the fibre during fixation hinders or prevents access of reducing agents. Padding with carrier and printing the discharge before fixation of the ground obviates this, but gives unlevel grounds. 13 Samaron (FH) disperse dyes applied either by carrier or by high temp. technique give equally good white and coloured discharges by steaming for 5–10 min. at 1·5–2·5 atm. pressure with several reducing agents (Rongalit H, Decrolin, Manofast). The discharge is improved by adding carrier (Remol TRD) and anthraquinone. The latter on illumination can form a red discoloration fugitive to both light and heat. Owing to dissolution in the polyester fibre, vat dyes differ in tone from their colours on cellulose (yellow → greener, scarlet → yellower, green → bluer and duller, brown → redder). Their lightfastness, lower than on cellulose, can be improved by heat treatment, which can also be used, on polyester fibres, for oxidation after soaping. A reducing aftertreatment is then sometimes required to improve rubbing fastness. S.M.J.

PATENTS

Pigment Printing or Padding
FBy *BP* 804,738

Prints or dyeings having excellent resistance to abrasion, high brilliancy, excellent fastness to washing, and an extremely soft handle are obtained by applying a paste or liquor containing pigment, a basic self-cross-linking polymer, and an aqueous emulsion of a copolymer having reactive groups capable of reacting with the basic polymer when heated, drying, and then baking. Thus a paste is made up of 30% aqueous pigment paste (50 parts), 40% copolymer aqueous emulsion from butadiene (30), styrene (30), butyl acrylate (30), and methacrylic acid (4). To this are added 70% aqueous emulsion of benzine (550), water (165), glycol (20), urea (30), and 30% aq. soln. (300 cps. at 25°c.) of the hydrochloride of the polymer from 0·3 mol. dipropylene-triamine and 1·0 mol. epichlorohydrin. Prints of excellent fastness on cotton are obtained by applying this paste, drying and baking for 5 min. at 140°c. C.O.C.

Discharge Effects on Cellulose Acetates of High Acetyl Value
BrC *BP* 805,740

Good white discharges which do not discolour on exposure to light or treatment with acidified bichromate or with Na percarbonate, are obtained on cellulose triacetate by use of discharge compositions containing thiourea dioxide (C.I. Reducing Agent 11), Zn thiocyanate, and salicylic acid. C.O.C.

Duplicating
W. Ritzerfeld and G. Ritzerfeld *BP* 805,272

A master sheet has an image formed on it by a composition containing an alkaline substance. A copy sheet is moistened with a solution of a non-staining diazo component and a coupling component, which react together only under alkaline conditions, in a volatile liquid. The two sheets are brought together so that transference of alkali from the master to the copy sheet causes the two components in the latter to couple to form a coloured monoazo image. C.O.C.

Colour Photography
International Polaroid Corpn. *BP* 804,974

A one-step photographic process for forming multicolour images and films used in the process are described. C.O.C.

3:3′:5-Trihydroxydiphenyl as the Azoic Coupling Component in Diazotype Material
Kalle & Co. *BP* 804,501

Prints obtained by using 3:3′:5-trihydroxydiphenyl in combination with azoic diazo components consisting of diazo compounds of *p*-phenylenediamines having a secondary or tertiary amino group and present as salts or double salts, have very strong dark brown tones and excellent contrast. C.O.C.

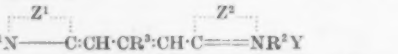
Photographic Colour Correction in Imbibition Printing
Comstock & Wemett *USP* 2,841,078

Preventing Dye Scum in Developing in Colour Photography
Ilford *BP* 805,174

Increase in the pH of the developing solution normally results in increased tendency to form dye scum, but addition of hydroxylamine or one of its salts effectively reduces or even eliminates dye scum. C.O.C.

Mixtures of Cyanine Dyes and Cadmium or Zinc Chloride or Bromide as Supersensitisers
Kodak *BP* 805,945

Dyes of formula—



(R¹, R², and R³ = Alk; X = acid radical; Z¹ and Z² = atoms to complete a heterocyclic nucleus of the thiazole, benzothiazole, naphthothiazole, oxazole, benzoxazole, naphthoazole, benzoselenazole, naphthoselenazole, or 2-quinoline series), e.g. 3:3′-dimethyl-9-ethylthiacarbocyanine bromide, have their photosensitising powers much increased if there is present 5–35 g./mol. silver halide of a chloride or bromide of Cd or Zn. C.O.C.

Coloured Images in Poly(vinyl Alcohol) Film
American Optical Co. *BP* 805,173

The film is first sensitised with tetrazotised *o*-dianisidine and then exposed to ultraviolet radiation through a cyan master colour separation positive, developing to form the corresponding cyan image in the film, and then after neutralising the coupler left in the film, repeating these processes using yellow and magenta master colour separation positives. C.O.C.

Multilayer Reversal Colour Photographic Material
Kodak *BP* 804,662

Printing or Decorating in Two or More Colours on Curved Ceramics, Glass, Plastics, etc.
Murray Curvex Printing *BP* 804,751

Mordants for Basic Dyes in Organic Colloid Layers (III p. 117)
Urea Glutarate as Printing Assistant (III p. 117)
Phthalogens (FBy) and Phthalocyanines (IV p. 121)
Anthraquinone Dye Developers (IV p. 123)
Dye Developers (IV p. 124)
Photographic Sensitisers containing more than one *mero*Cyanine Dye Radical (IV p. 126)

X—SIZING AND FINISHING

Rapid Contact Drying of Silk Fabrics
O. A. Kremnev, V. R. Borovskii, and A. A. Edzhubov

Tekstil. prom., **18**, 42–44 (July 1958)

The moisture content of silk fabrics can be reduced from 150–200% to 30% within 8 min. by passing the fabric over a 14-μ. thick aluminium foil acting as a heating element (86°c.). The foil is connected to the mains through a transformer. A detailed diagram of the drying table and the circuit is given. G.J.K.

Chlorine-Resistance of Drip-dry Resin Finishes
R. M. Reinhardt, L. W. Mazzeno, T. W. Fenner, and J. D. Reid

Amer. Dyestuff Rep., **47**, 505–508 (28 July 1958)

The effect of various curing conditions and catalysts on the stability to laundering of a cyclic ethyleneurea–formaldehyde (CEU) resin (Aerotex EU) at ~ 5% resin on a 3·2-oz. cotton fabric is reported. The treated fabrics were laundered in two types of domestic washing machine with water containing 0·1% chip soap and 0·2% available chlorine (from sodium hypochlorite) and ironed dry over 12 cycles. The approx. warp breaking strength of the untreated fabric fell from 44 lb. initially to 34 lb. after 15 washes compared with typical respective values of 27 and 26 lb. for all the treated fabrics. Crease resistance of the treated fabrics was well maintained throughout and there was no discoloration or loss of resin. In a similar comparison of a melamine (Resloom HP), a methylated melamine (Aerotex M3), a CEU and a CEU–acrylate–silicone resin, the first showed severe (0·15% retained chlorine after 1 wash), the second slight yellowing, while the two latter showed no change in appearance after 12 washes. The addition of the thermoplastic resins to CEU resin gives rise to somewhat better tear strength. E.C.

Textile Finishes

F. Fortess

Amer. Dyestuff Rep., **47**, P 513–P 522 (28 July 1958)

This review of recent literature (21 references), supported by original data discusses the functions of finishing agents and the site of action on or in the fibre. With wash–wear finishes for cotton or rayon, 1% cyclic ethyleneurea resin is sufficient to prevent solution of the fibre in cuprammonium solutions because of cross-linking of the cellulose molecules. This effect contributes to shrink-resistance while internal polymer formation from the resin gives dry and wet wrinkle-resistance and accounts for wash–wear properties.

The application of silicone finishes relies upon the ease of hydrolysis of alkyl silicon halides and the ease of oxidation of the silicon-hydrogen bond leading to cross-linking. Silicone finishes reside on the surface of the fibre as a tough, flexible hydrophobic lubricating envelope not chemically bonded to the fibre. Silicones confer water-repellence, tear- and abrasion-resistance, improved wrinkle recovery and reduction of needle-cutting and fusion on stitching due in many cases to the lower frictional coefficient of the treated fibres. Disadvantages are increased seam slippage, yarn shift, static generation, and susceptibility to oil staining. Data on some of these factors are tabulated.

Aqueous alkali treatments of polyester fibres remove the highly oriented surface layers, giving subdued lustre and improved softness and draping. Cellulose triacetate responds by hydrolysis of the surface layers to cellulose, leading to reduced static generation, better gas- and oxygen-fading resistance of the dyed fabric, softer handle, and less sticking on ironing. In contrast to secondary cellulose acetate, which hydrolyses very rapidly under alkaline treatment, cellulose triacetate can be treated with more latitude in control.

Trisaziridinylphosphine oxide developed for fireproofing cotton also causes cross-linking of cellulose molecules with attendant improvement in dimensional stability and wrinkle recovery. Diepoxides from epichlorohydrin and glycerol or other polyols are reactive to cellulose and confer wash–wear properties. Conventional epoxy resins have also been applied in textile treatment in conjunction with polyvinyl alcohol, epoxidised soya-bean oil, and a polyethylene dispersion. Diisocyanates although toxic in application may prove useful as textile finishing agents.

Various products derived from ethyleneimine, e.g. octadecylethyleneurea, are now available for use with melamine and urea resins to confer water-repellence and softness, and several fluorine-containing products, e.g. the chromium complex of perfluorocaprylamide and the perfluorobutyl ester of polyacrylic acid, are being applied to develop water- and oil-repellent properties especially on wool fabrics. E.C.

Flameproofing of Cellulose Fabrics. II—Action of Chloromethanephosphonic Acid and Urea on Cellulose Fabrics

R. Schiffner and G. Lange

Faserforsch. und Textiltech., **9**, 417–424 (Oct. 1958)

Cotton and cellulosic fabrics are treated with solutions of urea and chloromethanephosphonic acid, with solutions from melts of these components, and with solutions of such melts containing formaldehyde. Changes of strength and flame resistance, before and after rinsing, are determined, and the possible mechanism is briefly discussed. W.R.M.

Mothproofing of Wool with Dieldrin

M. Lipson and J. R. McPhee

Text. Research J., **28**, 679–686 (Aug. 1958)

Dieldrin does not react with wool, but some of the insecticide penetrates the fibres when applied from acidic aqueous emulsions. This small amount is sufficient to mothproof the wool and it is not removed by normal dryecleaning methods which do not cause swelling. It can be completely extracted, however, with hot methanol or acetone. An initial application of 0·05% on wt. of wool gives a mothproofing effect which will withstand many standard tests (ASTM Class 2), and machine washing for 9 hr. Dieldrin has been applied to large amounts of wool at various stages of processing, the results of the trials being given. The treatment should preferably be given to the last wet stage, since some of the insecticide is removed by subsequent dyeing, bleaching or milling. S.B.D.

Drip-dry Resin Finishes for White Cotton Fabrics

H. C. Borghetty

Amer. Dyestuff Rep., **47**, P 509–P 512 and P 523 (28 July 1958)

Performance requirements for drip-dry or wash–wear finishes and laundering tests therefor are discussed. AATCC 69–52 (Chlorine Damage) is too stringent a test, but AATCC 36–57 (Colour Fastness) resembles domestic drip-dry laundering. One commercial laundering is equivalent to twenty domestic drip-dry washes. Current wash–wear finishes are not adequate to withstand repeated commercial launderings, and a similar but not identical fall in crease-resistance and in wash–wear rating is observed under commercial laundering.

Diepoxides (not effective in crease-resistance) combined with cyclic ethylene urea–formaldehyde resins show the best retention of weft tensile strength in treated fabrics over 35 commercial launderings, with triazone resins second, but the wash–wear ratings are fair and poor respectively. Cyclic ethyleneurea- and urea-resin-treated fabrics are severely tendered after 10 and 5 launderings respectively. E.C.

PATENTS

Heating Filaments

E. B. Bates *BP* 805,522

Filaments are passed under controlled tension through a mass of hot metal balls or granules. C.O.C.

Permanent Sizing of Cellulosic Textiles with Rubbery Materials

B. F. Goodrich Co. *USP* 2,840,442

Excellent sizing and high abrasion resistance are imparted to cellulosic yarns by treating them with a butadiene–acrylic nitrile copolymer latex so as to form a discontinuous coating on the yarn. The abrasion resistant finish is fast to repeated launderings. The process is particularly applicable to cloths for boiler suits and the like. C.O.C.

Cyanoethylating Cotton with an Aqueous Emulsion of Sodium Hydroxide and Acrylonitrile

Institute of Textile Technology *USP* 2,840,446

Cotton treated with an aqueous emulsion of NaOH solution in acrylonitrile at 24–93°c. for 1–360 minutes so that the cotton contains 0·5–6·5% of N has its amorphous portion preferentially cyanoethylated while retaining all its crystalline structure. The aqueous phase of the emulsion is 50–150% on the weight of the cotton and contains 0·50–10·0% of NaOH, the ratio of NaOH to cotton being, by weight, 0·005 : 0·10. C.O.C.

Water Repellent Finish by use of an Aqueous Emulsion of a Silicone Resin

Deering Milliken Research Corpn. *BP* 804,509

The fabric is impregnated with an aqueous emulsion of an incompletely polymerised organo hydrogen polysiloxane and is then dried and baked to complete polymerisation of the polysiloxane. The emulsion contains an ammonium-salt of an organic acid as the emulsifying agent and in addition contains stannous, cuprous, or ferrous hydroxide. Thus Dow Corning Silicone Resin 1107 (a methyl hydrogen polysiloxane) (50 parts) and myristic acid (7·5) were dissolved in toluene (35) and perchloroethylene (9·5). This resin solution (20 lb.) was then added to an aqueous solution made up of water (969·5 lb.), $SnCl_2.2H_2O$ (2·5), and 28% aq. ammonia (8) at 85°F., a good emulsion being obtained by simple stirring. Cloth composed of wool and polyester fibre (45 : 50), impregnated with its own weight of this emulsion, dried and then baked for 10 min. at 200°F. was rendered highly water repellent. The water repellent finish was fast to drycleaning and to washing. C.O.C.

Reducing the Felting Power of Wool

Wool Industries Research Assocn. *BP* 804,781

The action of a protease on wool is much enhanced if the wool is first treated with a dilute aqueous organic peracid of formula R·CO·OO·H (R = H or Alk) preferably in presence of an anionic wetting agent. Thus wool hosiery fabric treated with dilute aqueous peracetic acid (12 parts in 3000) for 1 min. at 20°c. in presence of an anionic wetting agent, rinsed and then treated with papain and $NaHSO_3$ at pH 6·7 for 30 min. at 50°c. had very much less felting shrinkage than other samples treated with either peracetic acid or papain and bisulphite alone. C.O.C.

V— Photo-dehalogenation and -oxidation of *leuco*-Solutions

Ibid., 3353–3361

leuco-Solutions of the halogenated dyes Cibanone Brilliant Orange GK and Gold Yellow RK, and Caledon Brilliant Orange 6R, Orange 2RTS, and Brilliant Orange 4RN undergo rapid dehalogenation when exposed to visible light. In the presence of excess of dithionite, dehalogenation is complete, but, in catalytically prepared *leuco*-soln. with no excess of reductant, it proceeds to the extent of statistically one halogen atom per mol., which releases sufficient oxidant to oxidise completely and thus ppt. all the *leuco*-dye present. Thus, a dye containing n halogen atoms per mol. can undergo n successive reduction-irradiation-pptn. cycles. Potentiometric determinations show that not all the halogen liberated appears as halide ion; some is probably lost by adsorption on pptd. dye. Examination of the irradiation products by chromatography, etc., confirms the dehalogenation, and indicates that no monohalogenated product is formed from either di- or tri-halogenated dyes. The occasional production, under severe reducing conditions, of a *leuco*-soln. which was not rapidly pptd. on irradiation, an effect enhanced by pyridine, is ascribed to the formation of tautomeric oxanthranol. The spectral region responsible for photo-dehalogenation is located as approx. 450–650 mμ. for all five dyes.

VI— Coulometry of the Polarographic Oxidation of *leuco*-Dyes

Ibid., 3362–3367

The number of electrons involved in the polarographic oxidation of *leuco*-soln. of the eleven dyes of IV, anthanthrone, and Caledon Blue RSN, are determined micro-polarographically, and found to be 2 for all the cpd. except the five which are halogenated and undergo photo-dehalogenation-oxidation in *leuco*-soln.; for these, the apparent number of electrons is one. By conventional and differential absorption spectroscopy, the products of the exhaustive polarographic oxidation of the dihalogenated photo-active *leuco*-dyes are shown to contain ca. 25 moles % of the analogous unhalogenated dye. Both this partial dehalogenation and the low electron loss during electro-oxidation are accounted for by a tentative reaction scheme, in which a *leuco*-dye mol. adsorbed on the mercury-drop anode is dehalogenated by the joint action of mercury and water, either during or after electro-oxidation, yielding mercury halide and a hydroxyl radical, which then oxidise another *leuco*-dye mol. H.H.H.

Constitution of Remazol (FH) Dyes

H. Zimmermann

Melliand Textilber., **39**, 1026–1027 (Sept. 1958)

Remazol (FH) dyes such as dye $-SO_2\cdot CH_2\cdot CH_2\cdot O\cdot SO_3H$ react in alkaline medium with cellulose via vinyl residues giving cellulose ethers like cellulose$-O\cdot CH_2\cdot CH_2\cdot SO_2-$dye; hydrolytic side-reactions probably yield hydroxyethyl sulphones of little affinity. S.M.J.

Effect of Afterchrome and Vat Dyeing on Manufacture and Wear Characteristics of Wool

W. Hausmann

Melliand Textilber., **39**, 900–905 (Aug.), 1013–1017 (Sept. 1958)

Loose wool is dyed with afterchrome and vat dyes, spun, woven, and converted into garments. Part of the vat-dyed wool is aftertreated with an antibacterial agent (Eulan FL), part of the chromed wool is spun and woven differently. Six months' wearing trials of 80 suits dry-cleaned weekly show no significant differences, whereas manufacturing and mechanical properties appear superior for the vat-dyed wool. For this, dyeing is shorter, steam-consumption lower, though dyestuff costs are higher; in spinning yield, yarn strength and extension are higher; breakages in spinning and weaving are lower; less time is required in milling, but more in washing; wet and dry tear strengths, extensions (less markedly) and bursting strengths before and after abrasion are higher, though abrasion lowers the latter more than for chrome-dyed wool. The antibacterial treatment has little effect on the above properties, whilst different spinning and weaving improves the chrome-dyed wool, albeit not to the level of the vat-dyed wool. One year's weathering does not lower abrasion resistance; tear strength of vat-dyed cloth, though lowered more, is still better. S.M.J.

Reaction of Cellulose Fibres with Reactive Dyes and the Influence of Fibre Structure

K. Schwertassek

Faserforsch. und Textiltech., **9**, 321–329 (Aug. 1958)

Swelling and solution in sulphuric acid is used to study the reaction of cellulose fibres with cold-dyeing Procion dyes. Diminished solubility suggests cross-linking. Variations in structure may also be shown by this method. No effect on solubility was observed when hot dyeing Procion and Cibacron dyes were used. Sorption of iodine suggests blocking but not cross-linking. W.R.M.

Application of Procion (ICI) Reactive Dyes by Continuous Processes

R. A. Brindley and I. D. Rattee

Melliand Textilber., **39**, 1017–1022 (Sept. 1958)

The pad(with $NaHCO_3$)-dry system can employ (1) hot-flues, (2) steam-heated drying cylinders, or (3) fluid beds. In (1) the goods are cool (< 60°c. for air temp. 110°c.) until moisture content falls below saturation value— rapidly with rapid air displacement— when fixation, requiring 2–4 min., substantially ceases owing to insolubilisation of dye, especially in deeper colours. This can be counteracted by altering drying conditions, better by introducing some steam which reduces drying rate by ca. 20% and increases rate of fibre–dye reaction by 20–60%, best by incorporating solvents such as urea in the pad-liquor, which increase fixation by lowering rate of evaporation, raising temp. of goods, and delaying pptn., but also favour migration. (2) provide more rapid heat transfer and therefore higher wet goods temp. and faster (60–90 sec.) and more extensive fixation. Light goods react more rapidly but less extensively than heavy or raised fabrics. Passing fabric between cylinder and felt increases reaction rate and decreases migration rate. (3) dry and fix in 10–12 sec. The pad–steam process, best with high expressions, fixes pale colours in 10–30 sec. Intermediate drying enables greater depths and Procion H brands also to be fixed in 3 min. Padding first with dye, then with NaOH in brine, and steaming, if performed wet on wet, is restricted to very short caustic pad liquors, as in the Standfast molten metal machine, or to pale colours, because the dye bleeding into the brine, hydrolysed only to the monohydroxymonochlorotriazinyl derivative, can still react and cause ending; if wet on dry, can be used for all depths of cold-dyeing or "H" Procions, fixed by $\not< 15$ or $\not< 60$ sec. steaming. Fixation characteristics of the Procion range under all these conditions are illustrated in 10 tables and 4 graphs. Also discussed are removal of unfixed dye, combined dyeing and resin finishing, and the dyeing of loose viscose rayon. S.M.J.

Dyeing Furs with Oxidation Dyes

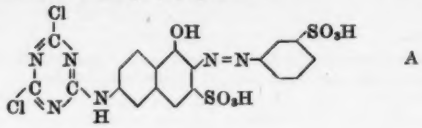
F. Brugger and L. Páris *Kožařství*, **7**, 354–7 (1957): *Chem. Abs.*, **52**, 19208 (10 Nov. 1958)

The conditions for the oxidation of *p*-phenylenediamine (C.I. 76060) by H_2O_2 in presence of $K_2Cr_2O_7$ were studied by determining the weight of insoluble oxidation products formed. Maximum catalytic effect was obtained with 0·05 oxidative equivalents of bichrome per C.I. 76060. Aqueous 3% bichrome was added to a solution of C.I. 76060 in a thermostat. Half the H_2O_2 (3% soln.) was added after 1 hr. and the other half after 2 hr. The temperature was maintained for a further 2 hr., after which the mixture was cooled overnight and the precipitate weighed. In presence of bichrome the maximum precipitate was formed with 3 equivalents of H_2O_2 (less precipitate with more H_2O_2), varying the temperature between 30–60°c. had no effect. In absence of bichrome, the precipitate increased with temperature, and the maximum precipitate obtained with 6 equivalents of H_2O_2 was only 25–50% of that obtained in presence of bichrome. Chromic salts have the same effect as bichrome, a significant point in the dyeing of lambskins tanned with chromic salts. In the absence of bichrome, addition of NH_4OH yields deeper dyeings, but the reverse is true in presence of bichrome. Dispersion of the precipitate is greater in presence of NH_4OH. When phenol is added to C.I. 76060, the weight of the precipitate increases but is not proportional to the weight of phenol added. The oxidation products of C.I. 76060 plus phenol contain less N, are less soluble and therefore more suitable as dyes than the oxidation products of C.I. 76060 alone. Pyrocatechol and resorcinol behave similarly to phenol but offer no advantages. C.O.C.

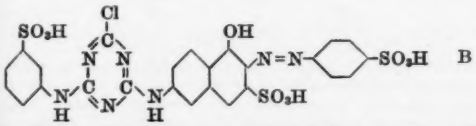
Reactive Dyes on Wool

R. Casty *S.V.F. Fachorgan*, **13**, 586–597 (Oct. 1958)

Reactive dyes consist mainly of dichlorotriazinyl (A) and monochlorotriazinyl (B) systems—



A



B

The former (much more reactive) belong to the cold-dyeing brands; the latter (less reactive) constitute the hot-dyeing class. Due to the reactivity of –Cl atoms in the triazinyl systems covalent bonds are formed between the dye and the substrate. The resulting dyeings are very fast to wet treatments. The fixation of reactive dyes on cellulose requires the addition of alkali. They are best applied to wool under mildly acid conditions. To avoid "skittery" dyeing the addition of certain cation-active assistants such as Neovadin AN (Ciba) is recommended. From a series of photomicrographs of fibre cross-sections, taken after 10, 20, 30, 40, 50, and 90 min. of dyeing (in the presence of Neovadin AN) it is apparent that although the dyebath was exhausted almost 100% after the first 30 min. of dyeing, the diffusion of the dye into the fibres did not commence until 10 min. later and was completed after 90 min. of total dyeing time (60 min. at the boil). It is possible that the anionic dye and cationic Neovadin AN form a complex which is initially deposited as a film round the wool fibres. When the bath is brought to the boil diffusion of the dye (or the complex) proceeds uniformly. Resulting dyeings are fast to rubbing, washing, potting, and light. The highest degree of fastness is obtained when surplus dye, which did not combine with wool, is removed by raising the pH of the bath to about 8 with ammonia or hexamethylene tetramine. Fastness properties on chlorinated wool are said to be as good as those on untreated wool. Reactive dyes are also of interest for Vigoureux printing of wool. L.A.T.

Dyeing Wool–Polyester Blends. IV— Effect of Dyeing Temperature

A. Würz *Melliand Textilber.*, **39**, 1023–1026 (Sept. 1958)

Fabrics were dyed at 94–100°C. for 1–8 hr. using a carrier— Palatinit M (BASF)— and yarns at 105–120°C. for 1 hr. using a dispersing agent— Uniperol W (BASF)— with 30 Palanil (BASF) disperse dyes. These differ appreciably in temp. sensitivity. Temperatures of 115°C. and 120°C. often give better results than carrier dyeing, unlike 105°C. and 110°C. Relative depths on both fibres are extensively tabulated. 7 of the dyes are recommended for 94°C., an additional 5 for 100°C. S.M.J.

Chemical Treatment of Polyamide Fibres with Pyridine Derivatives

S. Chrzczonowicz and B. Ostaszewski

I— The Use of Pyridine Derivatives as Assistants in Dyeing of Steelon (6-Nylon)

Zesz. Nauk. Politech. Lodz., No. 22 (*Chemia* 7), 47–56 (1958)

The possibility of the application of certain pyridine derivatives as assistants in the dyeing of Steelon, using acid and chrome dyes, has been examined. It was found that with acid dyes, pretreatment of the fibres with 2-(β-hydroxyethyl)-pyridine increased the depth of shade while at the same time causing some yellowing and precipitation of the dye in the dyebath. Poly-2-vinyl pyridine sulphate, used as a swelling agent with acid dyes, produced more uniform and intense dyeings on Steelon than on wool. The washing fastness was also improved. Quaternary derivatives of poly-2-vinyl pyridine used as surface-active agents in the dyeing with acid and chrome dyes produced improvement in fastness to washing to approximately the same degree as with Neolan Salt A (Ciba). The tensile properties of Steelon filaments remained unaltered as a result of the above pretreatments.

II— The Use of Quaternary Compounds Based on Poly-2-Vinyl Pyridine as Antistatic Agents

Ibid., 57–68

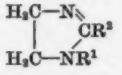
Preparations based on poly-2-vinyl pyridinium dimethyl sulphate were found to act as very efficient antistatic agents for continuous filament Steelon. Their effectiveness was increased by the addition of certain hygroscopic compounds such as glycerine, 2-(β-hydroxyethyl)pyridine, or electrolytes, e.g. NaCl, but not $CaCl_2$ due to formation of insoluble precipitates with Ca^{++} ions. The above preparations were stable over a wide range of temperatures and the treated yarn had long storage life under conditions which did not require strict control of temperature and humidity. L.A.T.

PATENTS

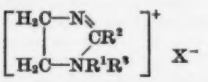
Level Dyeing of Nylon with Anionic Dyes in Presence of Substituted Imidazoline Compounds

Chemstrand Corpn. *BP* 806,026

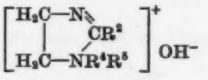
Level dyeings are obtained on nylon with direct, acid, premetallised acid, and afterchrome dyes applied from an aqueous acidic bath in presence of either a salt of an imidazoline of formula—



(R^1 = Alk, aralkyl, phenyl, hydroxyalkyl, or carboxyalkyl; R^2 = open aliphatic chain of 10–18 C), e.g. 1-butyl-2-hexadecylimidazoline, or a quaternary imidazolinium compound of formula—



(R^3 = same or different from R^1; X = Hal), e.g. 1:1-di-(2-hydroxyethyl)-2-heptadecylimidazolinium chloride, or a quaternary imidazolinium compound of formula—



(R^4 and R^5 = hydroxyalkyl, alkali metal-substituted hydroxyalkyl and alkali metal-substituted carboxyalkyl), e.g. disodium salt of 2-undecyl-1-(2-hydroxyethyl)-1-(carboxymethyl)-imidazolinium hydroxide. C.O.C.

Colouring Mineral Wool

American Rock Wool Corpn. *USP* 2,841,858

The fibres are coloured by adding a colorant, e.g. yellow iron oxide (C.I. Pigment Yellow 42), and a surface-active agent, to the annealing oil sprayed into the fibres at the entrance to the blow chamber in which they are collected. The resultant coloration is fast to rubbing and leaching. C.O.C.

Phthalogens (FBy) and Phthalocyanines (IV p. 121)

Polyamine Oxidation Dyes for Animal Fibres (IV p. 125)

Improving the Dyeability of Hydrophobic Polymers (VI p. 128)

Pigment Printing or Padding (IX p. 132)

IX— PRINTING

Recent History in Textile Printing

P. J. Wood

Amer. Dyestuff Rep., **47**, 755–756 (3 Nov. 1958)

Dyes for Photography— a Century of Progress

L. G. S. Brooker and P. W. Vittum

Proc. Perkin Centennial (Lowell, Mass.: AATCC. 1958), pp. 369–408:
J. Photographic Science, **5**, 71–88 (1957):
J.S.D.C., **74**, 765–767 (Nov. 1958)

Discharge Printing Trevira Polyester Fibre
K. Roth

Melliand Textilber., **39**, 1027–1030 (Sept. 1958)

Solution of dyes in the fibre during fixation hinders or prevents access of reducing agents. Padding with carrier and printing the discharge before fixation of the ground obviates this, but gives unlevel grounds. 13 Samaron (FH) disperse dyes applied either by carrier or by high temp. technique give equally good white and coloured discharges by steaming for 5–10 min. at 1·5–2·5 atm. pressure with several reducing agents (Rongalit H, Decrolin, Manofast). The discharge is improved by adding carrier (Remol TRD) and anthraquinone. The latter on illumination can form a red discoloration fugitive to both light and heat. Owing to dissolution in the polyester fibre, vat dyes differ in tone from their colours on cellulose (yellow → greener, scarlet → yellower, green → bluer and duller, brown → redder). Their lightfastness, lower than on cellulose, can be improved by heat treatment, which can also be used, on polyester fibres, for oxidation after soaping. A reducing aftertreatment is then sometimes required to improve rubbing fastness. S.M.J.

PATENTS

Pigment Printing or Padding
FBy *BP* 804,738

Prints or dyeings having excellent resistance to abrasion, high brilliancy, excellent fastness to washing, and an extremely soft handle are obtained by applying a paste or liquor containing pigment, a basic self-cross-linking polymer, and an aqueous emulsion of a copolymer having reactive groups capable of reacting with the basic polymer when heated, drying, and then baking. Thus a paste is made up of 30% aqueous pigment paste (50 parts), 40% copolymer aqueous emulsion from butadiene (30), styrene (30), butyl acrylate (30), and methacrylic acid (4). To this are added 70% aqueous emulsion of benzine (550), water (165), glycol (20), urea (30), and 30% aq. soln. (300 cps. at 25°c.) of the hydrochloride of the polymer from 0·3 mol. dipropylene-triamine and 1·0 mol. epichlorohydrin. Prints of excellent fastness on cotton are obtained by applying this paste, drying and baking for 5 min. at 140°c. C.O.C.

Discharge Effects on Cellulose Acetates of High Acetyl Value
BrC *BP* 805,740

Good white discharges which do not discolour on exposure to light or treatment with acidified bichromate or with Na percarbonate, are obtained on cellulose triacetate by use of discharge compositions containing thiourea dioxide (C.I. Reducing Agent 11), Zn thiocyanate, and salicylic acid. C.O.C.

Duplicating
W. Ritzerfeld and G. Ritzerfeld *BP* 805,272

A master sheet has an image formed on it by a composition containing an alkaline substance. A copy sheet is moistened with a solution of a non-staining diazo component and a coupling component, which react together only under alkaline conditions, in a volatile liquid. The two sheets are brought together so that transference of alkali from the master to the copy sheet causes the two components in the latter to couple to form a coloured monoazo image. C.O.C.

Colour Photography
International Polaroid Corpn. *BP* 804,976

A one-step photographic process for forming multicolour images and films used in the process are described. C.O.C.

3:3′:5-Trihydroxydiphenyl as the Azoic Coupling Component in Diazotype Material
Kalle & Co. *BP* 804,501

Prints obtained by using 3:3′:5-trihydroxydiphenyl in combination with azoic diazo components consisting of diazo compounds of *p*-phenylenediamines having a secondary or tertiary amino group and present as salts or double salts, have very strong dark brown tones and excellent contrast. C.O.C.

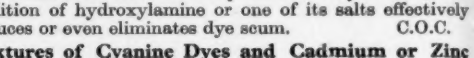
Photographic Colour Correction in Imbibition Printing
Comstock & Wescott *USP* 2,841,078

Preventing Dye Scum in Developing in Colour Photography
Ilford *BP* 805,174

Increase in the pH of the developing solution normally results in increased tendency to form dye scum, but addition of hydroxylamine or one of its salts effectively reduces or even eliminates dye scum. C.O.C.

Mixtures of Cyanine Dyes and Cadmium or Zinc Chloride or Bromide as Supersensitisers
Kodak *BP* 805,945

Dyes of formula—



(R^1, R^2, and R^3 = Alk; X = acid radical; Z^1 and Z^2 = atoms to complete a heterocyclic nucleus of the thiazole, benzothiazole, naphthothiazole, oxazole, benzoxazole, naphthoazole, benzoselenazole, naphthoselenazole, or 2-quinoline series), e.g. 3:3′-dimethyl-9-ethylthiacarbocyanine bromide, have their photosensitising powers much increased if there is present 5–35 g./mol. silver halide of a chloride or bromide of Cd or Zn. C.O.C.

Coloured Images in Poly(vinyl Alcohol) Film
American Optical Co. *BP* 805,173

The film is first sensitised with tetrazotised *o*-dianisidine and then exposed to ultraviolet radiation through a cyan master colour separation positive, developing to form the corresponding cyan image in the film, and then after neutralising the coupler left in the film, repeating these processes using yellow and magenta master colour separation positives. C.O.C.

Multilayer Reversal Colour Photographic Material
Kodak *BP* 804,662

Printing or Decorating in Two or More Colours on Curved Ceramics, Glass, Plastics, etc.
Murray Curvex Printing *BP* 804,751

Mordants for Basic Dyes in Organic Colloid Layers (III p. 117)
Urea Glutarate as Printing Assistant (III p. 117)
Phthalogens (FBy) and Phthalocyanines (IV p. 121)
Anthraquinone Dye Developers (IV p. 123)
Dye Developers (IV p. 124)
Photographic Sensitisers containing more than one *mero*Cyanine Dye Radical (IV p. 126)

X—SIZING AND FINISHING

Rapid Contact Drying of Silk Fabrics
O. A. Kremnev, V. R. Borovskii, and A. A. Edzhubov

Tekstil. prom., **18**, 42–44 (July 1958)

The moisture content of silk fabrics can be reduced from 150–200% to 30% within 8 min. by passing the fabric over a 14-μ. thick aluminium foil acting as a heating element (86°c.). The foil is connected to the mains through a transformer. A detailed diagram of the drying table and the circuit is given. G.J.K.

Chlorine-Resistance of Drip-dry Resin Finishes
R. M. Reinhardt, L. W. Mazzeno, T. W. Fenner, and J. D. Reid

Amer. Dyestuff Rep., **47**, 505–508 (28 July 1958)

The effect of various curing conditions and catalysts on the stability to laundering of a cyclic ethyleneurea-formaldehyde (CEU) resin (Aerotex EU) at ~ 5% resin on a 3·2-oz. cotton fabric is reported. The treated fabrics were laundered in two types of domestic washing machine with water containing 0·1% chip soap and 0·2% available chlorine (from sodium hypochlorite) and ironed dry over 12 cycles. The approx. warp breaking strength of the untreated fabric fell from 44 lb. initially to 34 lb. after 12 washes compared with typical respective values of 27 and 26 lb. for all the treated fabrics. Crease-resistance of the treated fabrics was well maintained throughout and there was no discoloration or loss of resin. In a similar comparison of a melamine (Resloom HP), a methylated melamine (Aerotex M3), a CEU and a CEU–acrylate-silicone resin, the first showed severe (0·15% retained chlorine after 1 wash), the second slight yellowing, while the two latter showed no change in appearance after 12 washes. The addition of the thermoplastic resins to CEU resin gives rise to somewhat better tear strength. E.C.

Textile Finishes

F. Fortess

Amer. Dyestuff Rep., **47**, P 513–P 522 (28 July 1958)

This review of recent literature (21 references), supported by original data discusses the functions of finishing agents and the site of action on or in the fibre. With wash–wear finishes for cotton or rayon, 1% cyclic ethyleneurea resin is sufficient to prevent solution of the fibre in cuprammonium solutions because of cross-linking of the cellulose molecules. This effect contributes to shrink-resistance while internal polymer formation from the resin gives dry and wet wrinkle-resistance and accounts for wash–wear properties.

The application of silicone finishes relies upon the ease of hydrolysis of alkyl silicon halides and the ease of oxidation of the silicon-hydrogen bond leading to cross-linking. Silicone finishes reside on the surface of the fibre as a tough, flexible hydrophobic lubricating envelope not chemically bonded to the fibre. Silicones confer water-repellence, tear- and abrasion-resistance, improved wrinkle recovery and reduction of needle-cutting and fusion on stitching due in many cases to the lower frictional coefficient of the treated fibres. Disadvantages are increased seam slippage, yarn shift, static generation, and susceptibility to oil staining. Data on some of these factors are tabulated.

Aqueous alkali treatments of polyester fibres remove the highly oriented surface layers, giving subdued lustre and improved softness and draping. Cellulose triacetate responds by hydrolysis of the surface layers to cellulose, leading to reduced static generation, better gas- and oxygen-fading resistance of the dyed fabric, softer handle, and less sticking on ironing. In contrast to secondary cellulose acetate, which hydrolyses very rapidly under alkaline treatment, cellulose triacetate can be treated with more latitude in control.

Trisaziridinylphosphine oxide developed for fireproofing cotton also causes cross-linking of cellulose molecules with attendant improvement in dimensional stability and wrinkle recovery. Diepoxides from epichlorohydrin and glycerol or other polyols are reactive to cellulose and confer wash–wear properties. Conventional epoxy resins have also been applied in textile treatment in conjunction with polyvinyl alcohol, epoxidised soya-bean oil, and a polyethylene dispersion. Diisocyanates although toxic in application may prove useful as textile finishing agents.

Various products derived from ethyleneimine, e.g. octadecylethyleneurea, are now available for use with melamine and urea resins to confer water-repellence and softness, and several fluorine-containing products, e.g. the chromium complex of perfluorocaprylamide and the perfluorobutyl ester of polyacrylic acid, are being applied to develop water- and oil-repellent properties especially on wool fabrics. E.C.

Flameproofing of Cellulose Fabrics. II—Action of Chloromethanephosphonic Acid and Urea on Cellulose Fabrics

R. Schiffner and G. Lange

Faserforsch. und Textiltech., **9**, 417–424 (Oct. 1958)

Cotton and cellulosic fabrics are treated with solutions of urea and chloromethanephosphonic acid, with solutions from melts of these components, and with solutions of such melts containing formaldehyde. Changes of strength and flame resistance, before and after rinsing, are determined, and the possible mechanism is briefly discussed. W.R.M.

Mothproofing of Wool with Dieldrin

M. Lipson and J. R. McPhee

Text. Research J., **28**, 679–686 (Aug. 1958)

Dieldrin does not react with wool, but some of the insecticide penetrates the fibres when applied from acidic aqueous emulsions. This small amount is sufficient to mothproof the wool and it is not removed by normal drycleaning methods which do not cause swelling. It can be completely extracted, however, with hot methanol or acetone. An initial application of 0·05% on wt. of wool gives a mothproofing effect which will withstand many standard tests (ASTM Class 3), and machine washing for 3 hr. Dieldrin has been applied to large amounts of wool at various stages of processing, the results of the trials being given. The treatment should preferably be given in the last wet stage, since some of the insecticide is removed by subsequent dyeing, bleaching or milling. S.B.D.

Drip-dry Resin Finishes for White Cotton Fabrics

H. C. Borghetty

Amer. Dyestuff Rep., **47**, P 509–P 512 and P 523 (28 July 1958)

Performance requirements for drip-dry or wash–wear finishes and laundering tests therefor are discussed. AATCC 69–52 (Chlorine Damage) is too stringent a test, but AATCC 36–57 (Colour Fastness) resembles domestic drip-dry laundering. One commercial laundering is equivalent to twenty domestic drip-dry washes. Current wash–wear finishes are not adequate to withstand repeated commercial launderings, and a similar but not identical fall in crease-resistance and in wash–wear rating is observed under commercial laundering.

Diepoxides (not effective in crease-resistance) combined with cyclic ethylene urea–formaldehyde resins show the best retention of weft tensile strength in treated fabrics over 35 commercial launderings, with triazone resins second, but the wash–wear ratings are fair and poor respectively. Cyclic ethyleneurea- and urea-resin-treated fabrics are severely tendered after 10 and 5 launderings respectively. E.C.

PATENTS

Heating Filaments

E. B. Bates *BP* 805,522

Filaments are passed under controlled tension through a mass of hot metal balls or granules. C.O.C.

Permanent Sizing of Cellulosic Textiles with Rubbery Materials

B. F. Goodrich Co. *USP* 2,840,442

Excellent sizing and high abrasion resistance are imparted to cellulosic yarns by treating them with a butadiene–acrylic nitrile copolymer latex so as to form a discontinuous coating on the yarn. The abrasion resistant finish is fast to repeated launderings. The process is particularly applicable to cloths for boiler suits and the like. C.O.C.

Cyanoethylating Cotton with an Aqueous Emulsion of Sodium Hydroxide and Acrylonitrile

Institute of Textile Technology *USP* 2,840,446

Cotton treated with an aqueous emulsion of NaOH solution in acrylonitrile at 24–93°c. for 1–360 minutes so that the cotton contains 0·5–6·5% of N has its amorphous portion preferentially cyanoethylated while retaining all its crystalline structure. The aqueous phase of the emulsion is 50–150% on the weight of the cotton and contains 0·50–10·0% of NaOH, the ratio of NaOH to cotton being, by weight, 0·005 : 0·10. C.O.C.

Water Repellent Finish by use of an Aqueous Emulsion of a Silicone Resin

Deering Milliken Research Corpn. *BP* 804,509

The fabric is impregnated with an aqueous emulsion of an incompletely polymerised organo hydrogen polysiloxane and is then dried and baked to complete polymerisation of the polysiloxane. The emulsion contains an ammonium-salt of an organic acid as the emulsifying agent and in addition contains stannous, cuprous, or ferrous hydroxide. Thus Dow Corning Silicone Resin 1107 (a methyl hydrogen polysiloxane) (50 parts) and myristic acid (7·5) were dissolved in toluene (35) and perchloroethylene (9·5). This resin solution (20 lb.) was then added to an aqueous solution made up of water (969·5 lb.), $SnCl_2 \cdot 2H_2O$ (2·5), and 28% aq. ammonia (8) at 85°F., a good emulsion being obtained by simple stirring. Cloth composed of wool and polyester fibre (45 : 50), impregnated with its own weight of this emulsion, dried and then baked for 10 min. at 200°F. was rendered highly water repellent. The water repellent finish was fast to drycleaning and to washing. C.O.C.

Reducing the Felting Power of Wool

Wool Industries Research Assocn. *BP* 804,781

The action of a protease on wool is much enhanced if the wool is first treated with a dilute aqueous organic peracid of formula R·CO·OO·H (R = H or Alk) preferably in presence of an anionic wetting agent. Thus wool hosiery fabric treated with dilute aqueous peracetic acid (12 parts in 3000) for 1 min. at 30°c. in presence of an anionic wetting agent, rinsed and then treated with papain and $NaHSO_3$ at pH 6·7 for 30 min. at 50°c. had very much less felting shrinkage than other samples treated with either peracetic acid or papain and bisulphite alone. C.O.C.

Water-repellent Finish

Midland Silicones *BP* 805,372

Organic fibrous material is rendered water-repellent by treating it with a composition comprising (1) a copolymer of 1–50 mol.% of siloxanes of formula $HOR^1{\cdot}SR^2_nO_{3-n/2}$ (R^1 = divalent satd. aliphatic hydrocarbon of > 2 C or a divalent satd. *cyclo*aliphatic hydrocarbon, the OH group being attached to a C atom at least 3 C atoms away from the Si atom; R^2 = monovalent hydrocarbon or halogenated monovalent hydrocarbon free from aliphatic unsaturation; n = 0, 1 or 2), and 99–50 mol% of siloxanes of formula $R^3_mH_pSiO_{(4-m-p)/2}$ (R^3 = monovalent hydrocarbon or halogenated monovalent hydrocarbon; m has an average value of 1–3; p = 0 or 1; $m + p = \not> 3$, and (2) a poly*iso*cyanate. Thus methyl hydrogen dichlorosilane (195·6 g.) and γ-acetoxypropylmethyldichlorosilane (646) were stirred into toluene (350) and water (376). After cohydrolysis was complete the copolymer solution was washed until neutral and the toluene solution concentrated to 70% by weight of siloxane. This solution (95) was diluted with toluene (95) and methanol (150). Benzene sulphonic acid was added as a catalyst and the solution kept at room temperature for 7 days. It was then washed with water and dried by azeotropic distillation until it weighed 95 g. This gave a solution of a copolymer of 15 mol.% of γ-hydroxypropylmethylsiloxane and 85 mol.% of methyl hydrogen siloxane. This solution (3 g.) and *m*-toluene di*iso*cyanate (1 g.) were dissolved in toluene (153 g.). Cotton cloth impregnated with this solution and air dried contained 1·2% by weight of combined silicone and di*iso*cyanate. The water-repellent finish given was not affected by laundering. C.O.C.

Flame-resistant Finish

Victor Chemical Works *USP* 2,841,507

A di-$\beta\gamma$-alkenyl β-cyanoethanephosphonate (e.g. that obtained by treating diallyl phosphite with acrylonitrile in presence of a Na alkenolate catalyst) is dissolved in an equal weight of solvent, e.g. $\beta\beta'$-dichloroethyl ether in presence of a polymerisation catalyst, e.g. benzoyl peroxide, and slowly heated until the solution's viscosity is 2–4 times its original value. The solution is then cooled and a solvent, e.g. ether or ethyl acetate, is added in which the partial polymer is insoluble. The partial polymer is separated and washed. It is then dissolved in a volatile solvent, e.g. methanol/ethylene dichloride 30:70 by vol. to a 25 to 50% solution. Fabric impregnated with this solution and heated at 100°C. for 30 min. is given a flame-resistant finish which is fast to washing. C.O.C.

Flame-resistant Fabrics

Greengate & Irwell Rubber Co. *BP* 805,499

Very good fire, flame, and glow resistance is given to natural or man-made fibres by treating them with a dispersion containing polyvinyl chloride plasticised with tritolylphosphate and ammonium phosphate, with or without addition of antimony oxide. C.O.C.

Bonded Fibre Fabric

Chicopee Manufacturing Corpn. *BP* 804,852

Dry powdered adhesive is distributed in a predetermined pattern in a carded lap. The adhesive is then softened and pressure applied to bond the fibres together following by hardening of the adhesive. C.O.C.

Leather-like Sheeting

B. F. Goodrich Co. *BP* 804,669

A non-woven fabric, e.g. a nylon mat, is impregnated with a solution of a polyester urethane, e.g. the reaction product of hydroxyl poly(ethylene adipate) and *p*-phenylene di*iso*cyanate, pressed, dried, aged, preferably in presence of a water-containing atmosphere, and pressed to form a leather-like material. The product may be embossed, stitched and otherwise treated and made up like leather. C.O.C.

Flame-resisting Agents for Cellulose (III p. 117)

Improving the Dyeability of Hydrophobic Polymers (VI p. 128)

Treating Polyethylene Terephthalate Fibres with Chlorinated Aliphatic Hydrocarbons (VI p. 128)

Coatings containing Tetrafluoroethylene Resin and Phenol-Aldehyde Resin (XIII p. 136)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Phase Structure of Cellulose

N. V. Mikhailov

J. Polymer Sci., **30**, 259–269 (July 1958)

The integral heats of solution of cellulose fibres of differing structure and molecular weight were determined in an aqueous solution of a quaternary ammonium base. The heat content of hydrated cellulose does not change over a wide range of orientation of the fibres, does not depend on the molecular weight of the cellulose, nor on the history of the fibre. The heats of solution change smoothly on transforming native cellulose into hydrated cellulose. This rules out phase differences in the structure of both forms, and in xanthogenated and regenerated cellulose, whose heats of solution were also examined. P.T.S.

Oxidation of Cellulose by Sodium Periodate and Sodium Chlorite

V. I. Ivanov, N. Ya. Lenshina, and V. S. Ivanova

Izvestiya Akad. Nauk S.S.S.R., otdel khim. nauk, 775–777 (June 1958)

Oxidation of cellulose by sodium periodate results not only in reaction at the α-glycolic grouping but also in the conversion of the $-CH_2OH$ group in the 6-position into –CHO and –COOH grouping. G.J.K.

Mechanicochemical Degradation of Cellulose during Vibratory Grinding

H. Grohn *J. Polymer Sci.*, **30**, 551–559 (July 1958)

Molecular-weight Distribution of Native Fibre and Wood Celluloses

M. Marx *J. Polymer Sci.*, **30**, 119–130 (July 1958)

Maxima between 6000 and 7000, and 8000 and 9000 for cotton, between 7000 and 8500, and 9500 and 11,000 for flax. Fractionation of the cellulose is carried out without degradation of the β-glucoside chains. Sprucewood cellulose also shows chain lengths up to 8000, and thus there is no essential difference between fibre and wood celluloses. P.T.S.

Spectroscopic Study of the Absorption of Metallic Cations by Oxidised Cellulose

I. N. Ermolenko and R. G. Zhbankov

Kolloidnŷĭ zhurnal, **20**, 429–435 (July–Aug. 1958)

The adsorption of cations from dilute solutions containing UO_2^{2+}, Pb^{2+}, Cu^{2+}, Cd^{2+}, Ca^{2+}, Ag^+, and Li^+ has been investigated by means of a previously developed infrared spectroscopic technique (*Izvest. Akad. Nauk Belorus. S.S.R., ser. teckhn. nauk*, 15 (1958)). It is found that changes in concn. have little effect on the degree of adsorption, indicating a high energy of adsorption. In contrast to cellulose and cellulose dialdehyde, the adsorption of UO_2^{2+}, Pb^{2+}, and Ca^{2+} on the carboxyl groups of oxidised cellulose shows a considerable increase in the 7–8 μ. region; the magnitude of adsorption depends upon cation nature. An ion exchange mechanism between the cation and the hydrogen of the carboxyl group is suggested. G.J.K.

Comparison of the Reactions during the Sulphite and Sulphate Cook

O. Samuelson

Svensk Papperstidning, **61**, 531–539 (15 Sept. 1958)

The author discusses and compares earlier work and reports his own experiments on the isolation and determination of aldonic acids in sulphite waste liquor. After removal of potassium, the liquor is passed through a strongly basic anion exchanger in sulphate form. The aldonic acids in the filtrate are taken up on a weakly basic anion exchanger in free base form, and eluated with dil. H_2SO_4. Excess sulphate ions are removed with Ba carbonate and the Ba salts of aldonic acids, mixed with other weak acids, are precipitated with alcohol. The Ba salts, which can be stored dry for a long period, are converted to the corresponding acids which are taken up on an ion exchanger in borate form and eluated with boric acid in the order: xylonic, arabonic, and hexonic acids. Separation of the latter is more difficult and is in the order: mannonic, gluconic, and finally galactonic acids. The presence in sulphite waste liquor of mannonic, xylonic, arabonic, gluconic, and galactonic acids was confirmed and the amounts of the three latter were determined. 29 references. R.A.

High-temperature Cellulose (Cellulose IV) by Heat-treatment of Mercerised Wood Pulp

A. Jullander and B. G. Rånby

Svensk Papperstidning, **61**, 680–685 (10 Oct. 1958)

When a rayon grade sulphite pulp is mercerised in 10% NaOH at 0°C. and heat-treated in 4% NaOH or glycerine at up to 170° and 280°C. respectively, there is a gradual decrease in water regain, accompanied by depolymerisation and transition into a new form, Cellulose IV. With caustic soda 50% conversion is attained at 175°C., while it is only reached at 200°C. with glycerine. Decrease in water regain is partly related to the depolymerisation, partly to lattice transition from Cellulose II into Cellulose IV. Because of the high temperatures required and the severe depolymerisation which occurs, this transition does not have any technical importance at present. R.A.

Distribution of Substituents in Partially Methylated Celluloses. III— Heterogeneous Reaction with Methyl Chloride

I. Croon

Svensk Papperstidning, **61**, 919–921 (15 Nov. 1958)

The reaction between alkali cellulose and methyl chloride is heterogeneous. Comparison of the distribution of substituents in partially methylated celluloses show that the relative reactivity at C_2 is greater for methyl chloride than for methyl sulphate. The ratio of rate constants is different for the two reagents and indicates that methyl chloride gives a more even distribution of substituents. Relative reaction rate constants for methyl chloride are: $k_2:k_3:k_6 = 5:1:2$. The etherification of cellulose depends not only on the structure of the alkali cellulose, but also on the nature of the reagent. R.A.

Heats of Wetting of Etherified Cellulose

E. F. Nekryach and A. V. Dumanskiĭ

Dokladÿ Akad. Nauk S.S.S.R., **121**, 136–137 (1 July 1958)

The calorimetric determination of the etherified (methyl, ethyl, and hydroxyethyl) cellulose from cotton linters and viscose rayon as starting materials is carried out. It is shown that the heat of wetting depends upon the degree of etherification and also on the chemical nature of the group. However, all these cellulosic ethers conform to a general pattern and show an increase in heat of wetting initially, a maximum, followed by a decrease with increasing degree of etherification; for cotton linters the maximum occurs at 8% etherification, whilst viscose rayon exhibits the maximum at 22%. Thus, an additional factor must be considered, i.e. the structure of the initial cellulose (pretreatment). G.J.K.

Formation of Cross-links in Cellulose during Nitration

H. Sihtola, B. Anthoni, and N.-E. Virkola

J. Polymer Sci., **30**, 1–4 (July 1958)

If cellulose is oxidised by periodate ions and then nitrated, it is incompletely soluble in acetone. The present work was undertaken to discover whether unoxidised cellulose, if nitrated in the presence of small difunctional molecules, became insoluble in acetone. Oxalic, glyoxylic, and sebacic acids were used. There is evidence that some cross-linking does take place with sebacic acid—with the other two difunctional molecules the evidence is equivocal. P.T.S.

Effect of the Regularity of Structure of Cellulose Triacetate on its Solubility and on the Properties of its Solutions

Z. A. Rogovin and D. L. Mirlas

Kolloidnŷĭ zhurnal, **20**, 376–381 (May–June 1958)

Gelatinisation of triacetylcellulose solutions in an acetylating mixture (acetic acid + acetic anhydride) in the presence of different catalysts (H_2SO_4, $HClO_4$, H_3PO_4) has been investigated. It has been shown that gelatinisation occurs when a cellulose triacetate with a high degree of regularity in its structure is formed. Gelatinisation is hindered when mixed esters are produced, e.g. by using a mixture of $HClO_4$ and HNO_3 as catalyst. G.J.K.

Ion-exchange Properties of Cellulose Phosphate

A. J. Head, N. F. Kember, R. P. Miller, and R. A. Wells

J.C.S., 3418–3425 (Oct. 19 58)

New Esters and Ethers of Cellulose

Z. A. Rogovin

J. Polymer Sci., **30**, 537–544 (July 1958)

The normal cellulose ester is prepared from cellulose alcoholate and methyl chlorocarbonate. *S*-Methyl chloromonothiocarbonate can be used to prepare the analogous thioester. A disulphide can be prepared by oxidation of the sodium salt of the thioester with iodine. Sodium phenoxide reacts with *p*-toluenesulphonyl phenyl esters of cellulose introducing a phenyl group into position 6. P.T.S.

Heavy-metal Complexes of Polysaccharide Formazans

L. Mester

J. Polymer Sci., **30**, 239–245 (July 1958)

Oxidised polysaccharides (cellulose, starch, etc.) will condense with phenylhydrazine. The condensates form intense red formazones with diazonium compounds. These, with heavy-metal salts, yield coloured metal complexes. Uranyl complexes show strong shading under the electron microscope, and the method may be developed for the study of polysaccharides. P.T.S.

Chitin. III— Hydroxyethyl and Ethyl Ethers of Chitin

S. N. Danilov and E. A. Plisko

Zhurnal obshcheĭ khimii, **28**, 2217–2223 (Aug. 1958)

The degree of etherification and solubility of hydroxyethyl ethers of chitin depend upon temp., reagent ratio, reaction time, the conditions of preparation of alkaline chitin, and also on the particle size of chitin. The ethyl ethers of chitin are soluble in alcohols, ketones, hydrocarbons, esters, and mixed solvents. Films prepared from ethylchitin compare favourably with those from ethylcellulose, giving, however, a reduced extension under the same load. G.J.K.

PATENTS

Internally Sized Paper

Hercules Powder Co. *BP* 804,504

Increased size retention is achieved by using a sizing mixture, at pH 5–9·5, comprising a ketone dimer of form $[RCH{=}C{=}O]_2$ containing 5–200% (by weight of dimer) of activated-clay, e.g. bentonite. R is alkyl (> 7 C), cycloalkyl (> 5 C), aryl, aralkyl or alkaryl. The amount of sizing mixture used should be so adjusted as to give a concentration of 0·05–2% by weight of sizing material based on the dry weight of the pulp. R.A.

Smoothing Coated Paper

Mead Corpn. *USP* 2,829,980

Adhesion to the smoothing roll is prevented and printability improved by maintaining the smoothing roll, which is covered with a film of release fluid (e.g. a mixture of ethylene glycol, water, and ethyl alcohol), at a temperature below the freezing point of the fluid coating, so effecting immobilisation or actual freezing of the blended and smoothed coating. The freezing point of the release agent must be below the temperature of the smoothing roll. R.A.

Carbon Paper with Improved Write-off Characteristics

Thilmany Pulp & Paper Co. *USP* 2,824,815

A non-film-forming chromium coordination complex of saturated perfluoromonocarboxylic acid with 4–10 C atoms in the molecule is applied to a sheet of basis weight between 4 and 15 lb. per ream, in an amount of between 0·02 and 0·5% of the weight of the sheet. The sheets are then dried and heated to convert the material in the liquid to the oleophobic and hydrophobic state, after which the sheet is coated with the usual wax-pigment composition. R.A.

XII— LEATHER; FURS; OTHER PROTEIN MATERIALS

Principles of Protein Structure and their Application to Synthetic Polypeptides and Fibrous Proteins

M. L. Huggins

J. Polymer Sci., **30**, 5–16 (July 1958)

A review of the evidence for the structures of α-keratin, β-keratin, polyglycine II, collagen. 53 references. P.T.S.

Review of the Processes for producing White Leather
V. Pektor and J. Ondráček
Kožařství, **7**, 287–297, 306–311 (1957): *Chem. Abs.*, **52**, 17771 (25 Oct. 1958)

The following processes are considered: (1) bleached chrome tannage, 1·7% Cr_2O_3, neutralised with $Na_2S_2O_3$, bleached with oxalic acid and bleaching syntans; (2) sulphochloride tannage preferably followed by Cr retan; (3) melamine tannage, which may follow a Cr pretannage; (4) Zr tannage. Al and HCHO tannages are only used in combination with other tannages. Fat-liquoring with sulphated castor oil and finishing with casein–resin binders and white pigments are recommended. C.O.C.

PATENTS

Retanning Mineral Tanned Leather with Resorcinol and Formaldehyde
Koppers Co. *USP* 2,840,445

Mineral tanned leather is soaked with water, excess water drained off, and is then drummed with 2–5% resorcinol (on the wrung out stored weight of the leather) and then with formaldehyde (1·5–3·0 mol. per mol. of formaldehyde), while maintaining the pH at 6·0–7·5 until all the resorcinol and formaldehyde have been absorbed by the leather. This yields leather of greater fullness, mellowness, and dimensional stability. C.O.C.

Resin Dispersions as Leather Finishes
ICI *BP* 805,492

A plasticised aqueous dispersion of a vinyl chloride–vinyl acetate copolymer is used as an anti-tack season coat over leather already treated with a resin-bound pigment finish, or the pigmented dispersion may be applied to crust leather as a finishing coat. The treated leather has a natural leather-like appearance with no tendency to stickiness and the finish is resistant to dry and wet rubbing. C.O.C.

XIII— RUBBER; RESINS; PLASTICS

Mechanism of Vulcanisation and the Action of Accelerators
B. A. Dogadkin
J. Polymer Sci., **30**, 351–361 (July 1958)

Elementary sulphur is not itself responsible for vulcanisation. The reaction proceeds *via* rubber polymer radicals and sulphur-containing radicals— perthiol and polysulphide radicals. P.T.S.

Some Rules Concerning Variations in the Melting Point in a Homologous Series of Hetero-chain Polymers
V. V. Korshak and T. M. Frunze
Dokladÿ Akad. Nauk S.S.S.R., **121**, 458–461 (21 July 1958)

The m.p. of various hetero-chain polyamides, obtained from diamines and acids containing both odd and even numbers of methylene groups, polyureates, polyurethanes, polyanhydrides, and polyesters have been determined. The authors show the presence of a linear dependence of the m.p. of the polymer on the hetero-chain characteristics; i.e. direct relationship between the physical properties and structure. Throughout the whole series of polymers investigated, the equation $y = Ax + 110$, where y = m.p., x = ratio in molar percentage of the number of linking groups (e.g. amide) to the number of methylene groups, and A varies between the limits 8·4 to − 3·6. G.J.K.

Determination of Specific Area and Pore Volume in Solid Polymeric Sorbents
A. A. Tager, M. V. Tsilipotkina, and A. I. Suvorova
Dokladÿ Akad. Nauk. S.S.S.R., **120**, 570–572 (21 May 1958)

A quantitative estimation of the degree of porosity of polymers based on the sorption of inert liquids has been attempted. Calculations are based on a modification of the fundamental equation for the S-type adsorption isotherm (C. Brunauer *et al.*, *J. Amer. Chem. Soc.*, **60**, 309 (1938)) and the following values for pore volume are reported— polystyrene 1·5% (of total polymer volume), cellulose 3%, and cellulose triacetate 8%. The specific area of polyvinyl alcohol is very small and agrees with that to be expected for non-porous sorbents. G.J.K.

Graft and Block Polymerisation
C. G. Overberger and A. Katchmann
Chem. Eng. News, 81–5 (10 Nov. 1958)

A review of the various ways of preparing graft and block polymers and of the use that has been made of these methods in industry. 10 references. C.O.C.

Kinetics of the Reactions of Thiourea with Formaldehyde. Reactions in Alkaline Media
K. Dusek
J. Polymer Sci., **30**, 431–458 (July 1958)

It was found possible to simplify the complex kinetic scheme and calculate rate constants. The equilibrium constants do not depend on pH. P.T.S.

Cationic Oligomerisation of the Stilbenes
D. S. Brackman and P. H. Plesch
J.C.S., 3563–3573 (Oct. 1958)

Oligomerisation (low polymerisation up to decamers) of *trans*-stilbene in benzene–hexane catalysed by titanium tetrachloride and trichloroacetic acid together is a cationic chain-reaction yielding a mixture of dimers and trimers which are subst. indanes, whereas in toluene there is a mixture of ~ 50% each of indane dimer and of 1 : 2-diphenyl-1-*p*-tolylethane; *cis*-stilbene forms the latter almost exclusively. In *n*-hexane at 0°c., *cis*-stilbene gives a mixture of oligomers up to and including a pentamer. H.H.H.

Rapid Polymerisation of Caprolactam
A. Hamann
Faserforsch und Textiltech., **9**, 351–361 (Sept. 1958)

In the rapid polymerisation of caprolactam with NaOH the actual initiator, sodium lactam, is formed in a preliminary equilibrium reaction, water formed in this reaction being removed. The polymerisation, in contrast to normal distributions, gives rise initially to a preponderance of large molecules, a normal distribution being obtained on reheating. W.R.M.

PATENTS

Aromatic Polyamines for Preventing Bloom on Rubber–Resins Blends
United States Rubber Co. *USP* 2,838,471

Use of 0·1–5·0% on the weight of the blend of a diprimary aryl amine, e.g. diaminodiphenylamine, prevents bloom without any adverse effect on the physical properties of the blend. C.O.C.

Organosiloxane Compositions which can be Rapidly Cured at Low Temperatures
Midland Silicones *BP* 804,199

A composition which can be cured in a few minutes at room temperature comprises (1) 0·01–5·0% by weight of a siloxane having > 2 units per molecule of formula $RHSiO$, the other remaining units being of formula $R_3SiO_{0.5}$ (R = monovalent hydrocarbon of < 76), (2) 99·99–95·0% of a hydroxylated polysiloxane of viscosity ≮ 100 cs. at 25°c., containing at least two silicon-bonded hydroxyl groups and an average of 1·9–2·0 monovalent hydrocarbon or halogenated hydrocarbon radicals of < 8 C, and (3) as catalyst a carboxylate of a metal or an organometallic compound. C.O.C.

Coatings containing Tetrafluoroethylene Resin and Phenol-Aldehyde Resin
DuP *BP* 804,178

Dispersions or solutions of tetrafluoroethylene resin and 5–100% (on the weight of the tetrafluoroethylene resin) of a thermosetting phenol-aldehyde resin, with if desired, addition of pigments, etc., yields coatings whose chemical and physical properties are as good as those of tetrafluoroethylene resin alone but which have much better adhesion to a wide variety of substitutes, e.g. metals, glass, textiles, etc. C.O.C.

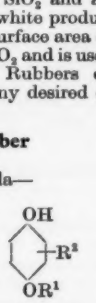
Fibrous Solid Silicon Monoxide— Reinforcing Pigment for Rubber
B. F. Goodrich Co. *BP* 804,992

Gaseous silicon monoxide is brought into contact with and condensed in presence of an inert gas containing a gaseous amine or NH_3 under non-turbulent conditions. This results in a fibrous, finely-divided silicon monoxide composition containing 1–8% by weight of N and having a large surface area. It is especially suitable as a reinforcing pigment for rubber. C.O.C.

Silicon Monoxide as a Reinforcing Pigment for Rubber
B. F. Goodrich Co. *BP* 804,993
Silicon monoxide free of a coating of an organosiloxane is a cheap reinforcing pigment for rubber. It should have an average particle size of 5–600 mμ. and a surface area of 60–300 sq.m./g. C.O.C.

Silicon Dioxide as a Reinforcing Pigment for Rubber
B. F. Goodrich Co. *BP* 804,994
Slowly heating finely-divided silicon monoxide under oxidising conditions to a temperature below the sintering temperature of Si and SiO_2 and at a rate insufficient to cause ignition yields a white product which retains all the original structure and surface area of the silicon monoxide. It consists entirely of SiO_2 and is used as a white reinforcing pigment for rubber. Rubbers containing it may be readily pigmented to any desired colour while still maintaining reinforcement. C.O.C.

Antioxidants for Rubber
ICI *BP* 805,368
Compounds of formula—



(R^1 = Alk; R^2 = tertiary hydrocarbon radical in *ortho* position to the OH or OR^1 and linked to the nucleus through a tertiary C atom of either a tertiary alkyl radical of 4–8 C or a 1-methyl*cyclo*pentyl, methyl-1-methyl*cyclo*pentyl, 1-methyl*cyclo*hexyl or methyl-1-methyl*cyclo*hexyl radical), e.g. the product obtained by reacting a 4-alkoxyphenol with a secondary aliphatic or *cyclo*aliphatic alcohol or the corresponding olefine in presence of a Friedel–Crafts catalyst, are superior to previous commercial antioxidants in combined anti-ageing, non-staining, and anti-flex cracking properties. C.O.C.

Unsaturated Ureido Compounds and Polymers and Copolymers Thereof
Rohm & Haas Co. *BP* 806,093
Monomers of formula CH_2 :CR^2·CO·X·A·NHCONHR^1 (R^1 = *tert.* Alk of 4–24 C, the *tert.* C being directly attached to the N atom; R^2 = H or CH_3; X = O or NH; A = alkylene group of 2–4 C, at least two of which extend in a chain between the adjoining X and NH radicals), e.g. *N*-β-methacryloxyethyl-*N'*-*t*-octylurea, are obtained by treating a compound of formula R^1NH_2 with an *iso*cyanate of formula CH_2:$C(R^2)$COOANCO. These monomers readily form polymers or copolymers which can be insolubilised by cross-linking, e.g. by heating to ≮ 175°C. to produce hard or rubbery films useful as protective coatings. C.O.C.

Improving the Dyeability of Hydrophobic Polymers (VI p. 128)
Coloured Images in Poly(vinyl Alcohol) Film (IX p. 132)
Determination of Molecular Weights and Dimensions by Light Scattering (XIV p. 138)

XIV— ANALYSIS; TESTING; APPARATUS

Analysis of Sodium Sulphide
ICI
J. Soc. Leather Trades Chem., **42**, 294–297 (Sept. 1958)
A soln. (I) of the sample in glycerol–water (5:100) is used for each phase of the analysis. Total sulphide may be determined iodometrically, either directly from I, or from ZnS pptd. from I by addition of a suspension of $ZnCO_3$. After successive treatments of I with soln. of NaOH, $BaCl_2$, Cd ammonium chloride, and N-HCl (to give a specified excess), with intermediate settling and filtration, the filtrate is titrated with N-NaOH. From the titre the NaHS and/or NaOH present in the original sample may be calc. Thiosulphate and sulphite contents are determined iodometrically on the filtrate after pptn. of S^{2-} as ZnS, as in the determination of total sulphide. As the method is not suitable for use with lime liquors it is not advanced as an official method of the S.L.T.C. J.W.D.

Regain Changes in Stretched Yarn
C. S. Clemson
J. Textile Inst., **49**, T 464–T 466 (Sept. 1958)
An apparatus for measuring the regain of stretched yarn by weight changes is described. Using viscose rayon, an extension of 15% at 65% R.H. will cause a 1·2% increase in weight after 140 min. because of the increased moisture content of the strained yarn. Several lower extensions were examined, and in general the regain increases with extension of the yarn. P.T.S.

Radiation Chemistry of Solutions. II— Dose Rate, Energy, and Temperature Dependence of a Leuco Triarylmethane Dosimeter Solution
W. A. Armstrong and G. A. Grant
Canadian J. Chem., **36**, 1398–1404 (Oct. 1958)
The title quantities have been investigated for the system 10^{-4} M-4:4'-(5-chloro-2-thenylidene)-bis-*NN*-dimethylaniline, 10^{-4} M. ferrous ammonium sulphate, 0·1 M. sodium chloride, and 7×10^{-3} M. hydrochloric acid. No energy dependence was observed in the range 0·16–2·0 Mev., and no dose-rate dependence was observed in the range 4–230 rads/min. However, with dose rates of 400–1200 rads/min., postirradiation dye fading was observed. In the dosage range 0–1000 rads this fading is not extremely serious. The system is slightly temp.-dependent below 30°C., but markedly dependent above. Dye yields of samples irradiated at 30°C. were 10% higher than those treated at 15°C., but 33% lower than those at 45°C. H.H.H.

Colorimetric Determination of *NN*-Diethyl-*p*-phenylenediamine in Colour Developers
B. A. Tsarev and V. V. Kol'tsov
Trudy Leningrad. Inst. Kino-Inzh. (3) 207–212 (1955): *Referat. Zhur., Khim.* Abstr. No. 37451 (1956): *Chem. Abs.*, **52**, 15316 (25 Sept. 1958)
Determination within ±3% of *NN*-diethyl-*p*-phenylenediamine (I) in developer in presence of different amounts of Na_2SO_3 is described. When I is oxidised in presence of a coupler the amount of dye formed depends on the concentration of Na_2SO_3 and on the ability of the coupler to compete with the Na_2SO_3. Of the couplers investigated 1-naphthol-3:8-disulphonic acid was more active than 1-naphthol-4:8-disulphonic acid, 1-naphthol-2-sulphonic acid and 1-phenyl-3-methyl-2-pyrazolin-5-one. The optimum amount of oxidising agent, length of oxidation, and reaction temperature which lead to formation of the maximum amount of dye are given. C.O.C.

Optical Measurements on Textiles
H. Köb
Textil Praxis, **13**, 932–936 (Sept. 1958)
After discussion of the limitations of some of the existing photoelectric instruments, the author gives an account of theoretical considerations which led to the development and design of a photometer for the measurement of the degree of whiteness of textile fabrics. The instrument has two photocells for measuring the reflected (W) and transmitted (T) light respectively. The percentage whiteness of the fabric $B\% = W\% + T\%$. It is said that the results of measurements obtained with this instrument are independent of both the thickness and the moisture content of the fabric. L.A.T.

Spectrophotometer and Spectrophotocolorimeter
F. Braun
Teintex, **23**, 648–657 (Sept. 1958)
The spectrophotometer Lv10 and the spectrophotocolorimeter Lv20 are described, and details of the commercial instruments given. S.R.C.

Penetration Test for Resistance to Pressure Dyeing
R. L. Wormell
J. Textile Inst., **49**, T 384–T 388 (Aug. 1958)
If a synthetic fibre is too thermoplastic it may be compressed during dyeing, e.g. by the action of hot liquors, into an impenetrable mass. A method of assessing the penetrability of fibres is described here. The fibres are cut mechanically into short lengths, blown by a current of air, and allowed to settle randomly— 5 g. of fibre in a ¾-in. tube, 4½ in. long. The time for 200 c.c. of a dyebath blank solution to penetrate is measured. The pressure is maintained constant with a mercury head. A modified Liebig condenser maintains the temperature of the dyebath blank solution during penetration. The method has been used with protein and non-protein fibres. P.T.S.

Determination of pH and Density of Raw-wool Extracts
G. Nitschke and R. Müller
Faserforsch. und Textiltech., **9**, 385–388 (Sept. 1958)
Factors affecting measurements of pH and density of raw wool extracts are considered and conditions for making such measurements suggested. W.R.M.

Assessment of Pleat Retention in Blended Fabrics
G. E. Chadwick
J. Textile Inst., **49**, T 409–T 410 (Aug. 1958)
This is a description of the method used at the Shirley Institute to measure the permanency of pleats to wetting, and to wet or dry mechanical treatments. The fabric is pleated in a standard way and the crease angle before and after wet or mechanical treatment is measured and taken as an assessment of pleat retention. Some typical results with graded wool–nylon blends are given. P.T.S.

Estimation of the Average Degree of Polymerisation (D.P.) of Natural and Regenerated Cellulose—II
O. Deschler
Textil Praxis, **13**, 927–932 (Sept. 1958)
Preparation and standardisation of the Cu–ethylenediamine (Cuene) reagent are described. Cotton (0·2 g./litre) and regenerated cellulose (1·0 g./litre) are dissolved in the Cuene reagent. The soln. is filtered through sintered glass, and the viscosity measured at 20 ± 0·1°C. using a capillary viscometer of the Ubbelohde type. Then—

$$\text{D.P.} = 8330 \times \frac{\eta_{sp}}{1 + 0{\cdot}29\eta_{sp}}$$

The specific viscosity (η_{sp}) is obtained from the formula—

$$\eta_{sp} = \frac{t - t_0}{t_0}$$

where t_0 = time of flow of solvent, and t = time of flow of soln. A useful parameter for comparative purposes is the so-called "damage factor" (s), calculated from Eisenhut's formula—

$$s = \frac{\log\left(\frac{2000}{x} - \frac{2000}{y} + 1\right)}{\log 2}$$

where x = D.P. of material before chemical treatment (e.g. bleaching), and y = D.P. of the same material after treatment. To obviate cumbersome calculations the value of s can be estimated directly from a nomogram. For normally bleached cotton $s \leqslant 0{\cdot}4$, and when $s \geqslant 0{\cdot}7$ the material is severely overbleached. L.A.T.

Action of Alkaline Iodine Solutions on Cotton Cellulose, Hydrocelluloses, and Periodate Oxycelluloses
R. L. Colbran and T. P. Nevell
J. Textile Inst., **49**, T 333–T 344 (July 1958)
The aldehyde groups in chemically modified cellulose are often estimated by measuring the amount of iodine consumed from an alkaline solution when the groups are oxidised. There are three main objections to this method: alkaline solutions of iodine are not stable; side-reactions consume an excess of the reagent; and some chain scission occurs in alkaline solution, causing more aldehyde groups to be formed in the cellulose or its derivatives. The effects of pH, temperature, and potassium iodide concn. on the stability of the solutions and their action with the cellulose materials have been examined, but the method has been shown to be unsatisfactory because these faults cannot be eliminated or allowed for. P.T.S.

Method of Testing Water-repellency of Fabrics permeable to Air using a Bundesmann-type Apparatus
Textile Institute
J. Textile Inst., **49**, P 255–P 266 (June 1958)
A description of interlaboratory trials designed to reduce the variability of results obtained using different machines. Control of water temperature and pH reduced the variability of water absorption, but no relationship was found between water temperature or water pH and penetration. The use of mechanical shakers reduces the between-machine variability. Combined with a mechanical shaker, steel wipers also reduce the between-machine variability. It has not been found possible to account for the differences which still exist between machines.
P.T.S.

Incorporation of Iodine in Crystalline Regions of Cellulose
K. Schwertassek and J. Doubek
Faserforsch. und Textiltech., **9**, 361–366 (Sept. 1958)
Studies are made of the penetration of iodine into the crystalline regions when more than 8–12% of iodine is absorbed, and the influence of such penetration on estimates of crystallinity by the iodine sorption method. Diagrams and a table are given relating the reduced iodine sorption to the percentage of amorphous material.
W.R.M.

Determination of Molecular Weights and Dimensions by Light Scattering
C. Ruscher and J. Dechant
Faserforsch. und Textiltech., **9**, 366–375 (Sept. 1958)
A summary of the theoretical basis of light scattering in solutions of macromolecules is given and an instrument for measuring the intensity of the scattered light is described. Results obtained with solutions of some polymers are discussed. W.R.M.

Recommended Procedure for using a Bundesmann-type Apparatus
Textile Institute
J. Textile Inst., **49**, P 254 (June 1958)
Using the procedure laid down in Textile Testing Standard No. 8 (1955) the variability between tests of water-repellency on a given machine can be reduced to a satisfactory level but the variation between different machines is large. P.T.S.

Polarographic Study of *p*-Nitrosodimethylaniline
V. N. Dmitrieva and V. D. Bezuglyĭ
Zhurnal obshcheĭ khimii, **28**, 2021–2028 (Aug. 1958)
The polarographic constants of *p*-nitrosodimethylaniline (I) are determined. Reduction of I at the dropping mercury electrode is dependent upon pH. Thus, two waves are observed, one in acid solution (pH 5·7) and the other in alkaline solution (pH 10·2). In unbuffered acidic solutions the two waves are observed simultaneously. The mechanism of the reduction of I under these conditions is investigated and it is shown that the transfer of four electrons occurs. This method is claimed to be of use for the quantitative determination of I. G.J.K.

Polarographic Determination of some Derivatives of 2-Ethyl-anthraquinone
L. M. Stifman, S. V. Syavtsillo, and L. G. Kuryakova
Zhurnal obshcheĭ khimii, **28**, 2028–2030 (Aug. 1958)
The half-wave potentials of 2-ethyl anthrone (I) and 2-ethyl-10-oxanthrone (II), using 0·6 M. tetramethyl ammonium bromide and aq. methanolic solutions containing a little benzene, have been determined and are shown to be 1·4 and 1·3 v. respectively (with respect to the saturated calomel electrode). Within the range 0·001–0·01 M. of I and II, a linear relationship exists between wave-height and concn. G.J.K.

Spectrophotometric Determination of Trace Molybdenum and Tungsten in Titanium Dioxide (C.I. Pigment White 27) with Dithiol
L. G. Stonhill
Chemist Analyst, **47**, 68, 69 (1958): *Chem. Abs.*, **52**, 18083 (10 Nov. 1958)
Traces of Mo and W in TiO_2 pigments are determined by first fusing with Na_2CO_3, grinding to powder, digesting with conc. HCl, adding water, passing SO_2 through to reduce Mo and extracting the Mo as the dithiol(toluene-3:4-dithiol) complex with amyl acetate. Absorbance of the green Mo-dithiol complex is obtained at 670 mμ. W is then determined by adding 20% $SnCl_2$ and conc. HCl to an aliquot of the aqueous extract in a separating funnel and then adding the dithiol in amyl acetate, warming to 50°c., shaking for 5 min. After three heating-shaking periods, determine the absorbance of the bluish green at 640 mμ. The concentrations of the two elements are obtained from standard curves. Cr and V in TiO_2 can be determined by a diphenylcarbazide colorimetric and an oxidation–reduction $FeSO_4$ titration, respectively.
C.O.C.

Investigation of Textile Damage
E. Kornreich
Textil-Rund., **13**, 458–464 (Aug. 1958)
Summary of tests previously published in British journals. C.J.W.H.

Polarographic Estimation of Thiol and Disulphide Groups in Wool

J. P. E. Human

Text. Research J., **28**, 647–654 (Aug. 1958)

Methods for determining thiol sulphur only and thiol plus disulphide sulphur in intact wool are described. In the first case, the wool is allowed to react with an excess of an organic mercury cpd. (either *p*-chloromercuribenzoic acid or neohydrin) in a buffer containing urea at pH 7–8·5, while in the second case sulphite as well as urea is present in a buffer at pH 8·5. In both, the excess organic mercury cpd. is determined polarographically. Two existing methods for the polarographic determination of thiol plus disulphide sulphur in protein hydrolysates, amperometric titration with mercuric chloride and the use of the catalytic wave produced by cysteine and cystine in the presence of cobalt ions, have been adapted for the special case of wool to cope with 10–100 mg. samples and 0·05–0·10 mg. samples respectively. An indication of the reliability of the non-hydrolytic methods is given.

S.B.D.

Modification of the Method for the Estimation of End Groups in Polyamides

E. Turska and L. Wolfram

Zesz. Nauk. Politech. Lodz., No. 22 (*Chemia* 7), 79–82 (1958)

The object of this investigation was to find a convenient laboratory procedure for estimation of acid end groups with a view to calculation of the number average molecular weights of polyamides. After critical study of the existing techniques the method of Waltz and Taylor (*Anal. Chem.*, **19**, 448 (1948)) was adopted for modifications to enable the titration to be carried out at room temperature instead of 175°c. The polyamide (0·15 g.) was dissolved in benzyl alcohol (20 ml.) by heating in a current of nitrogen at 150°c. After rapid cooling to 20°c. and the addition of *n*-propanol (3 ml.), the soln. was titrated with KOH, using phenolphthalein as indicator. The authors claim that this procedure gives as reproducible results as any of the existing methods. L.A.T.

Evaluation of Formaldehyde Pretreatment for Wool Bleaching

J. E. Moore and R. A. O'Connell

Text. Research J., **28**, 687–690 (Aug. 1958)

The formaldehyde treatment of wool fibres and fabric prior to bleaching with hot alkaline peroxide liquors is investigated. Although pretreated fabrics exhibit lower alkali solubility and better handle after bleaching, the characteristic degradation, shown by various fibre and fabric properties, is not retarded by the pretreatment. Formaldehyde treatment alone under the conditions used causes some degradation, but this is unlikely to be severe enough to cause concern for many fabric uses. Other experiments without the pretreatment fail to support the claim that wool fibres possess increased susceptibility to damage when strained prior to bleaching. S.B.D.

Chemical Basis of Human Colour Vision

W. A. H. Rushton

Research, **12**, 478–483 (Dec. 1958)

A discussion of recent work to determine the mechanism of human colour vision and how the different types of colour blindness can arise. C.O.C.

Determination of the Solubility of Dyes

M. Capponi

Amer. Dyestuff Rep., **47**, 673–674 (6 Oct. 1958)

Translation from German— cf. J.S.D.C., **74**, 327 (April 1958). C.J.W.H.

Diffusion and Viscosity Measurements on Macromolecules in Solution

C. Rossi, U. Bianchi, and V. Magnasco

J. Polymer Sci., **30**, 175–186 (July 1958)

The determination of precise information about molecular dimensions and the interaction between polymer molecules and solvent molecules from diffusion and viscosity measurements is the object of the work. For high polymers, measurements must be carried out in an "ideal" solvent. The ideal solvent will consist of a mixture of two components, one a solvent and the other a non-solvent (precipitating agent). The proportions of one to the other at any temperature can be determined viscometrically. P.T.S.

Electrophotometric Colorimetry as an Objective Method for Measuring the Concentration of Acid Dyes

V. V. Kozlov and R. R. Solntseva

Sbornik Nauch. Rabot., Moskov. Inst. Narod. Khoz. im. G. V. Plekhanova (ii), 220–237 (1957): *Chem. Abs.*, **52**, 17717 (25 Oct. 1958)

A Ts 3–A photometer with Se photocells and a set of light filters was used to measure the strength of solutions of C.I. Acid Blue 92, C.I. Acid Red 26, C.I. Acid Yellow 11, C.I. Acid Orange 7, C.I. Acid Red 1, C.I. Acid Red 71, and C.I. Acid Black 24. The electrophotometric method gave more accurate results than the visual colorimetric method. Results obtained with the photometer differed from those obtained by chemical analysis by 0·2–0·5% for blue and orange, 1% for red, and 1·5% for yellow dyes. In the colorimetric determination permissible error is 5%. The highest sensitivity was achieved at an optimum concentration for blue, green, and red of up to 0·01 g./l., and for yellow up to 0·02 g./l. A calibration curve prepared with a standard dye should be used for analyses of commercial samples. Presence of another dye ingredient (> 10%) makes photometric determination impossible. C.O.C.

Colour Fastness to Daylight at High Humidity— Proposed New Test

Society of Dyers and Colourists Light Fastness Subcommittee

J.S.D.C., **74**, 756–758 (Nov. 1958)

Weathering Fastness Assessment— A Review of the Present Position

K. McLaren

J.S.D.C., **74**, 759–762 (Nov. 1958)

The difficulties encountered in devising a test for the determination of fastness to weathering are described, as well as the attempts made to overcome them. No immediate solution is in sight, but the problem is being jointly studied in England, France, Germany, and Switzerland. AUTHOR

Measuring Crease and Pleat Sharpness and Retention by the Creasometer

P. R. Wilkinson and P. W. Ireland

Text. Research J., **28**, 674–679 (Aug. 1958)

The Creasometer is used to measure the geometry of a crease. This measurement, made on a pressed fabric which has been given a simulated wear or wash test, or both, can be converted to % crease retention and used to predict field performance. In addition, a measurement can be obtained of the ability of a fabric to be pleated by any of the available pressing methods. S.B.D.

Characterisation of Viscose Solutions— Determination of Gel Particles in Viscose

E. E. Treiber

Svensk Papperstidning, **61**, 794–802 (10 Oct. 1958)

A survey. Physico-chemical methods for the characterisation of viscose are described, together with available methods for making gel particles visible and recording them. The Boryniec–Kiessig method of counting gel particles is discussed in detail. 38 references. R.A.

Determination of Calcium in Cellulose by means of Ethylenediaminetetra-acetic Acid

H. Tötterman, M. Johanson, A. Kakko, and J. E. Martelin

Paperi ja Puu, **40**, 501–507 (Oct. 1958)

Three new indicators— Calcein, Cal-red, and Calcon— were tested at the Central Laboratory for Cellulose Industry, as murexide was not found satisfactory. Pulps of different origin and different content of Ca, Mg, Mn, Fe, and Cu were used. Both Calcein and Cal-red are superior to murexide, while Calcon is unsuitable for the complexometric determination of Ca in pulp. Cal-red gives a sharper colour change and better reproducibility than Calcein. R.A.

Analysis of Amino–Formaldehyde Resins

J. C. Morath and J. T. Woods

Anal. Chem., **30**, 1437–1440 (Aug. 1958)

Methods for the analysis of textile resins of the amino–formaldehyde type are reviewed and summarised and, in some instances, are given in detail. Resin components discussed are urea, ethyleneurea, thiourea, melamine, total and free formaldehyde, formaldehyde as hydroxymethyl, methylated and methylenic formaldehyde, and total and free methanol. L.T.W.

Detection of Benzenedicarboxylic and Benzoic Acids in Alkyd Resins by Infrared Absorption Spectrometry
M. L. Adams and M. H. Swann
Anal. Chem., **30**, 1322–1324 (Aug. 1958)

The three benzenedicarboxylic acids and benzoic acid can be detected in alkyd resins by examination of the infrared absorption spectrum of the dried resin film. Absorption characteristics in the 11–15 μ. region by which each acid may be detected in presence of each or all of the other acids are illustrated and tabulated. L.T.W.

Spectroscopic Detection of Silicon in Organic Silicon Compounds
J. Radell, P. D. Hunt, E. C. Murray, and W. D. Burrows
Anal. Chem., **30**, 1280–1281 (July 1958)

In a qualitative and semi-quantitative method for determining silicon in organosilicon compounds, the sample is suspended in acid solution and decomposed by spark discharge. Silicon gives emission bands at 6347 and 6371 A. L.T.W.

Silicone Detection
G. von Finck
Melliand Textilber., **39**, 1262 (Nov. 1958)

Water-repellent silicones are transferred with conc. H_2SO_4 to the glass of a test-tube, which then repels the hot acid, unlike fluorides even after washing. 50–100 mg. of fabric or one drop of extraction residue covered with 1·5 ml. conc. H_2SO_4 heated in $\gg$ 1·5 min. to the boil is inspected every 5 sec. After 20–40 sec. silicones cause the acid to run down the walls very rapidly, instead of wetting them homogeneously. Repetition after cooling, discarding the acid, washing and reheating with fresh acid confirms. 0·5–1 mg. polysiloxane is detectable. Silicic esters do not react. S.M.J.

PATENTS

Observing Moving Fabrics
Lindauer Dornier *BP* 803,843

The fabric is passed at full width over a rotating cylinder made of glass or the like, the speed of the fabric and of rotation of the cylinder being the same. The cylinder has a transparent shell or else an opaque shell having a window of transparent material corresponding in size to the section viewed by video cameras one or more of which are carried inside the cylinder and placed so that they scan the window. Lighting equipment also carried in the cylinder illuminates the fabric passing over the window. The video cameras are connected to television receivers placed so closely together that they reproduce a coherent integral image of the whole of the fabric on the window.

BP 804,099

The camera can be placed with its optical axis in any convenient direction if optical deflecting means, e.g. mirrors or prisms, are fixed in the line of sight of the camera so that the fabric passing over the window is viewed by the camera. C.O.C.

Preparation of Microscope Test Slides for Counting of Particles
Coal Industry (Patents) *BP* 805,732

Test Fungi in Textile Testing (VI p. 126)
Empirical Relations of Crease Recovery and Time (VI p. 127)
Maximum Alkali-combining Capacity of Wool (VI p. 127)

XV— MISCELLANEOUS

Informative Labelling of Textiles
O. Viertel
Textil Praxis, **13**, 1019–1025 (Oct. 1958)

In view of the variety of textile fibres with differing physical and chemical properties the author stresses the need for informative labelling of textiles which would convey, with the aid of signs and symbols, pictorial instructions for correct washing, dry-cleaning, and ironing. After a critical survey of present-day arrangements in several European countries (including Great Britain) the author makes certain proposals and advocates their universal acceptance. A washtub symbolises domestic washing treatment, an iron depicts pressing, and a ringed capital letter— A for all the usual dry-cleaning solvents, P for perchlorethylene or benzine, F for benzine only— signifies the method of dry-cleaning. In the case of articles which should not be washed, dry-cleaned, or ironed the appropriate symbol is cancelled by crossing. As an additional aid for better appreciation of instructions by the general public use is made of a three-colour scheme— green when wide choice of conditions of treatments is allowed, orange when certain limitations are imposed, and red when particular care is required. L.A.T.

PATENTS

Metallised Azo Dyes for Staining Wood
Glidden Co. *USP* 2,820,712

The chromium complexes of dyes of formula—

$$[X{\cdot}A{\cdot}N{:}N{\cdot}B](SO_2CH_3)_n$$

(A = benzene; B = radical of a phenolic, naphtholic or enolic coupling component containing an OH group *ortho* to the azo group; X = OH or COOH *ortho* to the azo group; $n = 1$ or 2), e.g. C.I. Acid Black 58 and C.I. Acid Brown 46, are soluble in many organic solvents. Such solutions when used for staining wood give exceptional penetration, clarity of colour and fastness to light. They are especially suitable for use in non-grain-raising stains and lacquers. C.O.C.

Combined Stain and Filler for Wood
Glidden Co. *USP* 2,820,711

A paste containing (*a*) "inert" solids, with or without pigments, which serve to fill the pores of the wood, (*b*) water-soluble dye of good fastness to light, (*c*) a film-forming material, and (*d*) a solvent to unite the whole, quickly stains and fills wood uniformly to a clear, clean colour without raising the grain. When diluted for use it is stable for at least 8 hr. and even if separation occurs on long standing, simple stirring restores it to its initial state. The treated wood does not blister nor does the filler lift when a mild lacquer sealer is applied. C.O.C.

Wood Stain and Filling Composition
Chadeloid Corpn. *USP* 2,817,594

The product obtained by coupling a citrus flavanone derivative, e.g. hesperetin, naringenin or eriodictyol, with an aromatic amine, yields a product which is highly effective as an aqueous stain for wood. C.O.C.

Removing Excess Colour from Fruit Juice or Wine
Rohm & Haas Co. *USP* 2,832,688

Treatment with an enzyme preparation containing an anthocyanase and then heating to $>$ 150°F. to destroy the enzyme is used. Suitable enzyme preparations can be prepared from a variety of fungi. C.O.C.

Enhancing and Preserving the Colour of Meat
Swift & Co. *USP* 2,830,907

Propionic acid and its edible esters when used with ascorbic acid have a synergistic effect in stabilising the colour of fresh meat, the protective effect lasting much longer than if either acid were used alone. C.O.C.

Removing Vinyl Chloride–Methyl Acrylate Co-polymers from Cellulosic Fibres
Chicopee Manufacturing Corpn. *USP* 2,832,663

The fibres are heated in a dilute caustic solution containing a little of a low mol.wt. aliphatic ketone or alcohol, e.g. methyl ethyl ketone and methyl alcohol, at above the b.p. of the solution for $\gg$ 5 hr. C.O.C.

Restoration of Faded Ink Writing
L. H. Almy and C. E. Hultman *USP* 2,833,612

The writing is treated with an aqueous solution of $Na_4(or\ K_4)Fe(CN)_6$, an acid, an alkali metal halide and an immiscible soluble alkaline earth salt, e.g. the document is immersed for 45 sec. in a solution at 45°c. containing water (7570 ml.), HCl s.g. 1·19 (15 ml.), NaCl (383 g.), $CaCl_2{\cdot}2H_2O$ (383 g.) and $K_4Fe(CN)_6{\cdot}3H_2O$ (24 g.) and then immediately rinsed in water for 45 sec. and dried. This restores the brilliance of the writing without blurring its edges or discolouring the paper. C.O.C.

Preservation of Colour in Canned Green Vegetables
Patent Protection Corpn. *USP* 2,827,382

Addition of a dibasic alkali metal or alkaline earth metal salt of glutamic acid to keep pH 8·0–8·5 after canning preserves the colour of the vegetables. C.O.C.